Advance Journal of Food Science and Technology 7(3): 144-153, 2015
DOI:10.19026/ajfst.7.1283
ISSN: 2042-4868; e-ISSN: 2042-4876

Submitted: September 07, 2014 Accepted: September 20, 2014 Published: February 05, 2015

Research Article

Optimization for Ultrasound-microwave Assisted Extraction of Pectin from Jujube Waste using Response Surface Methodology

[1]Fengqi Bai, [1, 2]Jie Wang and [1]Jie Guo
[1]College of Food Science and Technology, Agricultural University of Hebei,
[2]Engineering Technology Research Center for Processing of Agricultural Products, Hebei Province,
Baoding 071000, China

Abstract: Optimization of conditions for Jujube pectin extraction was investigated using Response Surface Methodology (RSM). Extraction parameters which are employed in this study are Liquid-Solid Ratio (LSR) (5-15), pH (1.5-2.5), ultrasonic time (10-20 min) and microwave irradiation time (40-60 sec) and they were optimized using a four factor three levels Box-Behnken response surface Design (BBD) coupled with desirability function methodology. The results showed that, all the process variables have significant effect on the yield of pectin. The satisfactory conditions for Jujube pectin extraction were obtained as follows: 10.03 mL/g of LSR, 1.97 of pH of sulfuric, 17.66 min of ultrasonic time and 52.73 sec of microwave irradiation time. Among the studied factors, microwave irradiation time had the greatest influence on yield. Under these conditions, the experimental yield of Jujube pectin was 1.95±0.06%, which is well in close agreement with the value predicted by the model.

Keywords: Box-behnken, *jujube* pectin, Response Surface Methodology (RSM), ultrasound and microwave assisted extraction

INTRODUCTION

Ziziphus jujube dates, commonly called Chinese date, whose cultivation originated in China, is a species of *Ziziphus* in the buckthorn family (*Rhamnaceae*) and it is indigenous to China with a history over three thousand years. There are about 135,0000t waste *jujube* every year for weather or insect pest and so on. A large quantity of dates (about 30% of the total production) is lost during picking, manufacture of some date products, or storage of second category dates which are generally discarded or partially integrated in animal feed (Besbes *et al*., 2006). Studies concerning the use of these by-products to develop new products are scarce and concern metabolites or biomass production (Abou-Zeid *et al*., 1991). It is discarded indiscriminately into the environment and thereby constituting environmental challenges. These waste jujubes are not presently being utilized for any value added process due to limited research activities focusing on the possible conversion of the waste to the other valuable products thereby making it available for dumping as solid waste. Therefore, the development of new, environmentally and friendly method to manage that problem is great concern. Dates contained about 2.27% of pectin. Since, the conversions of waste jujube into valuable by product such as pectin offers great scope for utilization and also creates more income for farmers, food processors and more importantly reduce environmental impacts of the waste.

Pectin is a family of complex polysaccharides present in cell walls of all land plants. The main components of pectin are polysaccharides in which (1→4) linked α-D-galacturonates and methyl esters predominate (Westereng *et al*., 2008). Pectin has a long safe history of use as gelling agent, thickener and stabilizer in food industry. Otherwise, pectin has been attempted to develop as drug delivery system (Liu *et al*., 2005), flocculant (Ho *et al*., 2010) and biosorbent. Commercial pectin is extracted from apple pomace and citrus peel at high temperature in the present of acid. Recent years, ultrasound-microwave assisted method (Fishman *et al*., 2006; Prakash Maran *et al*., 2013b, 2014; Rodriguez-Jasso *et al*., 2011; Seixas *et al*., 2014) which can distinctly reduce extraction time and energy has been employed for natural products extraction. In the aspect of pectin extraction, ultrasound-microwave assisted extraction can prominently increase yield, heighten the degree of esterification and acetylation. Recently, extraction and characterization of pectin and the relationships between factors and yield were studied (Faravash and Ashtiani, 2007; Sahari *et al*., 2003; Urias-Orona *et al*., 2010). In fact, all the investigations using full factorial design are

Corresponding Author: Jie Wang, College of Food Science and Technology, Agricultural University of Hebei, West Campus, No. 2596, Lekai South Street, Baoding, Hebei Province 071000, China, Tel.: +86-312-7528199; Fax: +86-312-7528189

RSM, which is a collection of statistical and effective way for the desired purpose. Most of these studies were concerned with the extraction of pectin using conventional heating, from which effect of variables on yield, properties and structure had been obtained. Few if any of these studies were directed toward understanding of combined effects of processing variables on the yield of ultrasound-microwave assisted extracted jujube.

In this research, the aim of this study was to look for the experimental conditions leading to the maximum pectin extraction from waste jujube. As many factors can influence the extraction yield, Response Surface Methodology (RSM) was applied to fit and exploit a mathematical model representing the relationship between the variables (LSR, pH of sulfuric acid, ultrasonic time and microwave irradiation) and the responses (yield) and obtained satisfactory conditions for extraction using Box-Behnken experimental design.

MATERIALS AND METHODS

Materials: The Traditional Chinese Jujube was obtained from Changzhou in Hebei Province and was disintegrated to 6 mm using disintegrator and was stored in sealed plastic bags at 20.0±1°C. Other chemicals, including Hydrochloric acid (HCl), Sulfuric acid (H_2SO_4), Carbazole ($C_9H_{12}N$) and D-L galacturonic acid ($C_7H_{12}O_6$), were analytical grade and supplied by Sigma.

Ultrasonic and microwave assisted extraction of pectin: The extraction process was carried out in microwave oven reaction system (Wang *et al*., 2007) (WP700, Galanz and Foshan, China). A 200 mL Pyrex beaker containing a mixture of 10.00 g of accurately weighed jujubes with different amount distilled water (50-150 mL) containing different pH values (1.5-2.5) were placed in the middle of the ultrasonic equipment exposed different time (10-20 min), then extraction process took place in the microwave equipment at the power of 560 W for irradiation times 40-60 sec. After the reaction, the extracts were allowed to cool down, filtered using filter paper (Whatman, No. 1), then supernatant was precipitated with an equal volume of 95% (v/v) ethanol. The coagulated pectin mass was washed with 95% (v/v) ethanol for two times to remove the mono and disacchardies. After the extraction, the pectin was subjected to drying at 55°C in the hot air mathematical techniques, has been proved to be an oven until it attains a constant weight. The Pectin Yield (PY) was calculated from the following equation:

$$PY = \frac{m_0}{m} \times 100\% \tag{1}$$

where,
m_0 : Weight of pectin
m : Weight of jujube

Box-behnken design: Response surface methodology was applied to determine the conditions of pectin yield of jujube. On the basis of the preliminary single factor experiment, a Box-Behnken Design (BBD) with three independent factors (X_1, liquid-solid ratio; X_2, solution pH; X_3, ultrasonic time X_4, microwave irradiation time) set at three variation levels was carried out (Table 1). And +1, 0, -1 encoded factors represent variables (Ni and Zeng, 2010). The yield of pectin was selected as the responses for the combination of the independent variables shown in Table 2. The correspondence between the coded and uncoded values can be obtained using the following equation:

$$x_i = \frac{X_i - X_0}{\Delta X_i} \tag{2}$$

where, x_i is the coded value of the variable, X_i is the corresponding actual value of variable, X_0 is the actual value of X_i at the centre point and ΔX_i is the increment of X_i. The experimental design consisted of 29 experiments including 29 factorial points and 5 replicates at the central point (Table 2). Each condition was carried out in triplicate and the mean values were stated as experimental responses. Moreover, all the experiments were randomized to minimize the effects of unexpected variability on the experimental responses.

Experiment design and statistical analysis: Experimental dates were analyzed using Design-Expert 8.0.6.1 (State-Ease Inc. Minneapolis, MN, USA) statistical package. A Box-Behnken response surface experiment design with 4 factors was to optimize and investigate the individual and interactive effects of process variables on the pectin yield of jujube. The experiments were conducted in a randomized order and the data were analyzed by multiple regression analysis in order to develop an empirical second order

Table 1: Box-behnken central composite experimental design factors and levels of encoded values

	Symbols		Levels		
Factors	Coded	Uncoded	-1	0	1
Liquid-solid ratio (mL/g)	x_1	X_1	5	10	15
Solution pH	x_2	X_2	1.5	2.0	2.5
Ultrasonic time (min)	x_3	X_3	10	15	20
Microwave irradiation time (sec)	x_4	X_4	40	50	60

Table 2: Box-behnken design with the observed responses and predicted values for the yield of pectin

Run	LS ratio (X_1 (mL/g))	pH (X_2)	Ultrasonic time (X_3 (min))	Irradiation time (X_4 (sec))	Observed (Y_0)	Predicted (Y)
1	0	0	0	0	2.013	1.964
2	-1	0	0	-1	1.268	1.242
3	0	0	0	0	2.017	1.964
4	1	0	0	1	1.562	1.534
5	0	0	0	0	1.942	1.964
6	0	0	0	0	1.990	1.964
7	-1	0	0	1	1.711	1.664
8	0	0	-1	-1	1.266	1.215
9	0	0	-1	1	1.682	1.692
10	0	1	0	1	1.506	1.462
11	0	-1	0	1	1.620	1.647
12	-1	-1	0	0	1.649	1.630
13	0	0	1	1	1.737	1.819
14	1	1	0	0	1.351	1.401
15	1	0	-1	0	1.390	1.361
16	1	0	1	0	1.859	1.809
17	0	1	1	0	1.703	1.693
18	0	-1	1	0	1.796	1.701
19	0	1	0	-1	1.131	1.127
20	0	-1	0	-1	1.138	1.206
21	0	1	-1	0	1.311	1.352
22	0	0	0	0	1.857	1.964
23	-1	0	1	0	1.622	1.674
24	0	0	1	-1	1.500	1.521
25	1	0	0	-1	1.187	1.180
26	-1	0	-1	0	1.616	1.689
27	1	-1	0	0	1.303	1.366
28	0	-1	-1	0	1.654	1.609
29	-1	1	0	0	1.363	1.331

regression polynomial mathematical model, which exhibits the relationships between response and independent variables. The construction and analysis of the experimental design, Analysis of Variance (ANOVA) to obtain the interaction between the process variables and the response, quality of the fit of the polynomial model (coefficient of determination (R^2), adjusted coefficient of determination (adj-R^2) and predicted coefficient of determination (pre-R^2) and optimization of process condition were obtained using Design-Expert 8.0.6.1. After optimization, triplicate experiments were performed under the optimal conditions and the average value of the experiments was compared with the predicted values of the developed model equation. All experiments were performed at least in triplicate and results were expressed as means of ±S.D. (Standard Deviation). Data obtained were analyzed using one-way Analysis of Variance (ANOVA, $p<0.05$, SPSS, version17.0). And p-value of <0.05 were considered to be statistically significant.

RESULTS AND DISCUSSION

Statistical analysis and model building: Twenty nine experiments were carried out according to the conditions indicated in Table 2. Response values (yield of pectin) were reported in the last column of this table. Regression analysis (Table 3) was made to the experimental data aiming at an optimal region for the responses study. The analyses of variance were used to determine the coefficient of determination, lack of fit and the significance of the linear, interaction effects and quadratic of the independent variables on the response.

The significance of each coefficient was determined using the F-test and p-value in Table 3. The p-value represents the significance of the corresponding coefficients in terms of the yield of pectin, with a smaller p-value indicating more significant impact of the corresponding coefficient. The results of regression coefficient analysis showed that the variable with the largest effect was the quadratic term of microwave irradiation time (X_4^2), followed by quadratic term of pH (X_2^2), liquid-solid ratio (X_1^2) and liner term of microwave irradiation time (X_4), the interaction effects of s liquid-solid ratio and ultrasonic time (X_1X_3), the interaction effects of liquid-solid ratio and pH (X_1X_2) and the quadratic term of ultrasonic time (X_3^2), the ultrasonic time (X_3), pH (X_2), which were extremely significant ($p<0.01$). Also, the interaction effects of liquid-solid ratio (X_1) were significant ($p<0.05$). However, the interaction effects of liquid-solid ratio and (X_1X_4), pH and ultrasonic time (X_2X_3), pH and microwave irradiation time (X_2X_4), ultrasonic time and microwave irradiation time (X_3X_4) were not significant ($p>0.05$).

An empirical quadratic polynomial model corresponding to the BBD was fitted to correlate the relationship between independent variables and the responses to predict the optimized conditions. The quadratic model is following as:

Table 3: Analysis of Variance (ANOVA) for response surface quadratic model for the extracted pectin (the yield of pectin) and independent variables (X_1, X_2, X_3, X_4)

Source	Coefficient estimate	S.S.	Degree of freedom	M.S.	F-value	p-value
Model	0.1964	0.0193	14	0.0014	27.2904	<0.0001
A-LS	-0.0048	0.0003	1	0.0003	5.4893	0.0344
B-pH	-0.0066	0.0005	1	0.0005	10.3555	0.0062
C-CST	0.0108	0.0014	1	0.0014	27.7523	0.0001
D-WBT	0.0194	0.0045	1	0.0045	89.2229	<0.0001
AB	0.0083	0.0003	1	0.0003	5.5076	0.0342
AC	0.0116	0.0005	1	0.0005	10.6110	0.0057
AD	-0.0017	0.0000	1	0.0000	0.2331	0.6367
BC	0.0062	0.0002	1	0.0002	3.0766	0.1013
BD	-0.0027	0.0000	1	0.0000	0.5651	0.4647
CD	-0.0045	0.0001	1	0.0001	1.5697	0.2308
A^2	-0.0244	0.0039	1	0.0039	76.1542	<0.0001
B^2	-0.0288	0.0054	1	0.0054	106.3846	<0.0001
C^2	-0.0087	0.0005	1	0.0005	9.6814	0.0077
D^2	-0.0315	0.0064	1	0.0064	127.3112	<0.0001
Residual		0.0007	14	0.0001		
Lack of fit		0.0005	10	0.0001	1.1949	0.4680
Pure error		0.0002	4	0.0000		
Cor. total		0.0200	28			
Std. dev.		0.0071		R-squared	0.9647	
Mean		0.1577		Adj R-squared	0.9293	
C.V. %		4.5101		Pred R-squared	0.8336	
Press		0.0033		Adeq precision	16.3515	

S.S. : Sum of square; M.S.: Mean square

$$Y = 0.20 - 0.005X_1 - 0.007X_2 + 0.011X_3 + 0.019X_4 + 0.008X_1X_2 + 0.012X_1X_3 - 0.002X_1X_4 + 0.006X_2X_3 - 0.003X_2X_4 - 0.004X_3X_4 - 0.024X_1^2 - 0.029X_2^2 - 0.009X_3^2 - 0.032X_4^2 \quad (3)$$

where, Y is the predicted response (the Yield of pectin) and X_1, X_2, X_3, X_4 are coded values of liquid-solid ratio, solution pH, ultrasonic time and microwave irradiation time, respectively.

The analysis of variance (F-test) shows that the second order model matches well with the experimental data. The Coefficient of Variation (CV) indicates the degree of the precision. Here, a lower value of CV (4.51) indicates the experiments are more precise and reliable (Prakash Maran *et al*., 2013a). The precision of a model can be represented by the determination coefficient (R^2). The determination coefficient (R^2) implies that the sample variation of 96.47% for the yield of pectin is attributed to the independent variables. Adjusted coefficient of determination (adj-R^2) and predicted coefficient of determination (pre-R^2). Meanwhile, the high R^2 (0.965), adj-R^2 (0.929) and pre-R^2 (0.834) clearly demonstrated that the experiment and the theoretical values predicted by polynomial model had a very close agreement. From the analysis, the F-value of 27.29 and p-value<0.001 indicates the response surface quadratic model was significant. Furthermore, results of the ANOVA indicated that the lack of fit of 0.468 was insignificant.

Analysis of response surface:

Perturbation plot: In generating the perturbation plots, all the factors were plotted on the same response graph. This graph could be used to find factors that most affect the response. A steep slope or curvature in a factor shows that the response is sensitive to that factor. A relatively flat line shows insensitivity to change in that particular factor (Gupta and Ako, 2005). The response (Y) was plotted against the deviation from the reference point by changing only one factor over its entire range while holding all other factors constant as shown in Fig. 1. From the Fig. 1, it was observed that, pH and liquid-solid ratio have great influence on the extraction yield compared with ultrasonic time and microwave irradiation time.

The relationship between the responses and the experimental variables can be illustrated graphically by plotting three-dimensional response surface plots (Fig. 2a to f).

Effect of liquid to solid ratio on the yield of pectin: The yield of pectin was increased by raising the liquid-solid ratio from 5-15 (Fig. 2a to c). The increasing ratio of liquid-solid indicates that bigger liquid-solid ratio firstly led to higher yield of pectin. Further expanding the liquid-solid ratio, however, almost had no significant effect on the yield of pectin. Water can absorb microwave energy leading to enhance water penetrating the cell of jujube, which promotes the contact surface area between the jujube and water. As a result, it is easy for the release of pectin from jujube cell into the surrounding solvent resulting in raising the yield of pectin to liquid-solid ratio of 10.33 (Liu *et al*., 2006). However, higher ratio would decrease the microwave adsorption of jujube, because more energy was absorbed by water which stops the penetration of pectin into the solvent and decreases the yield of pectin.

Effect of pH on the yield of pectin: In order to select a proper pH to enhance the yield of pectin, studies were carried out at different pH (1.5-2.5). From the results, it

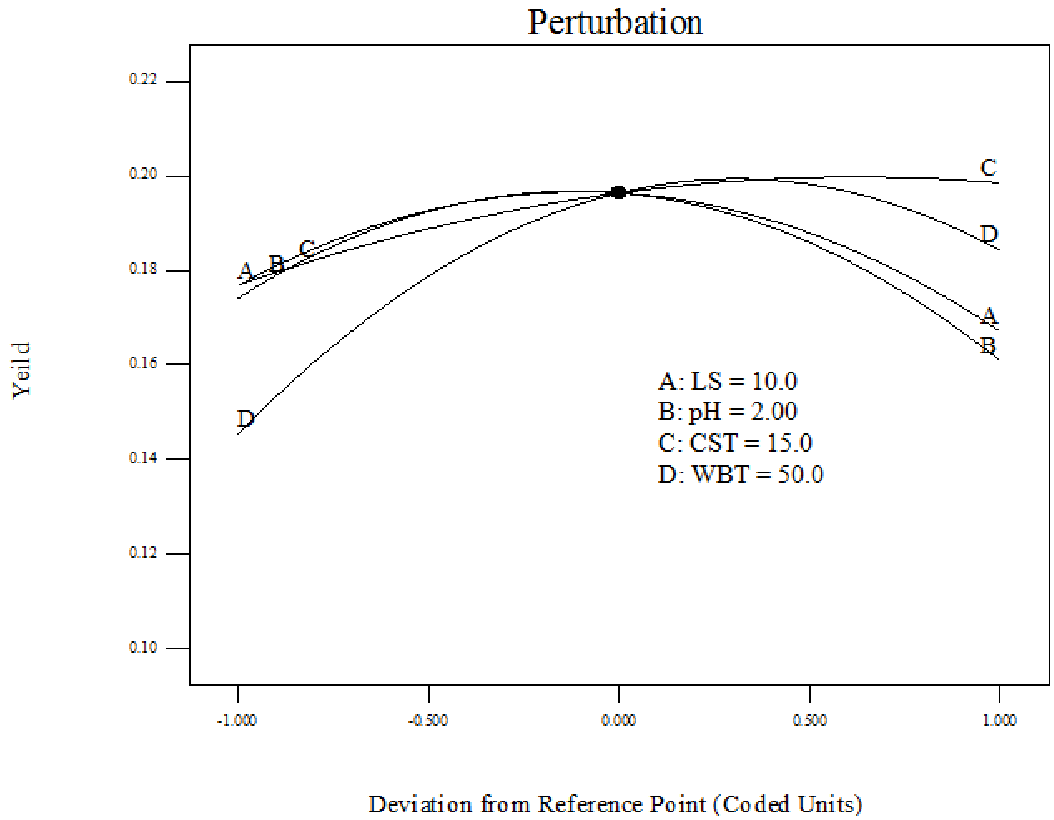
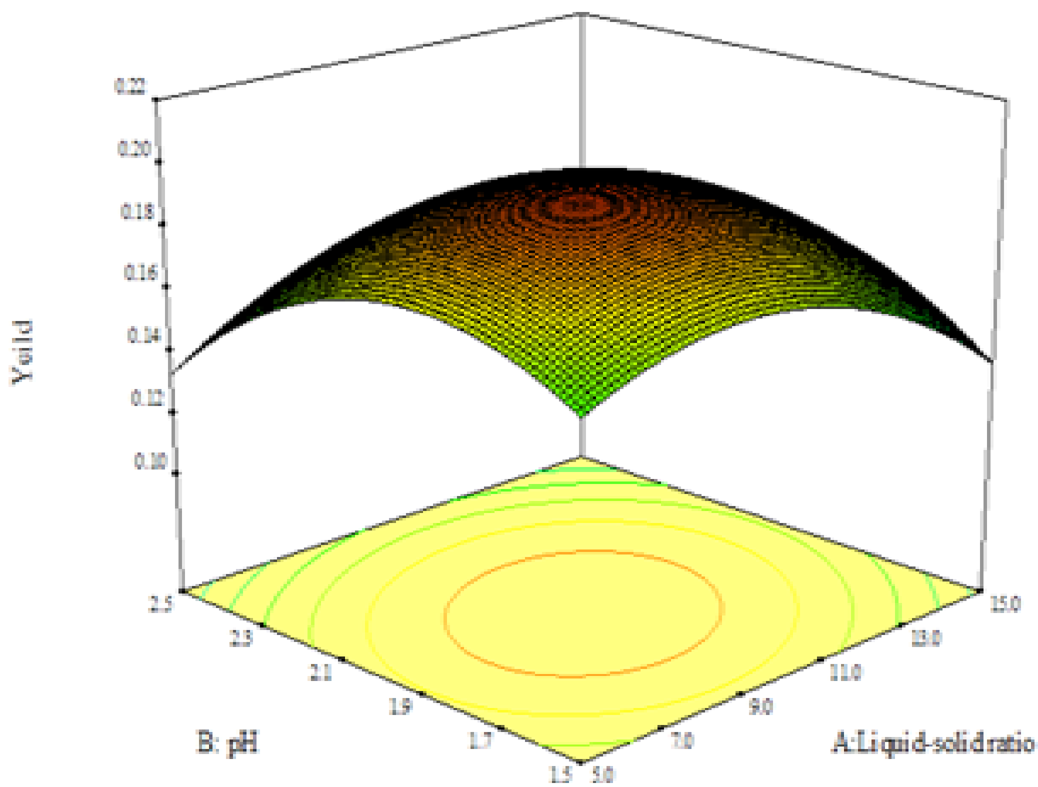
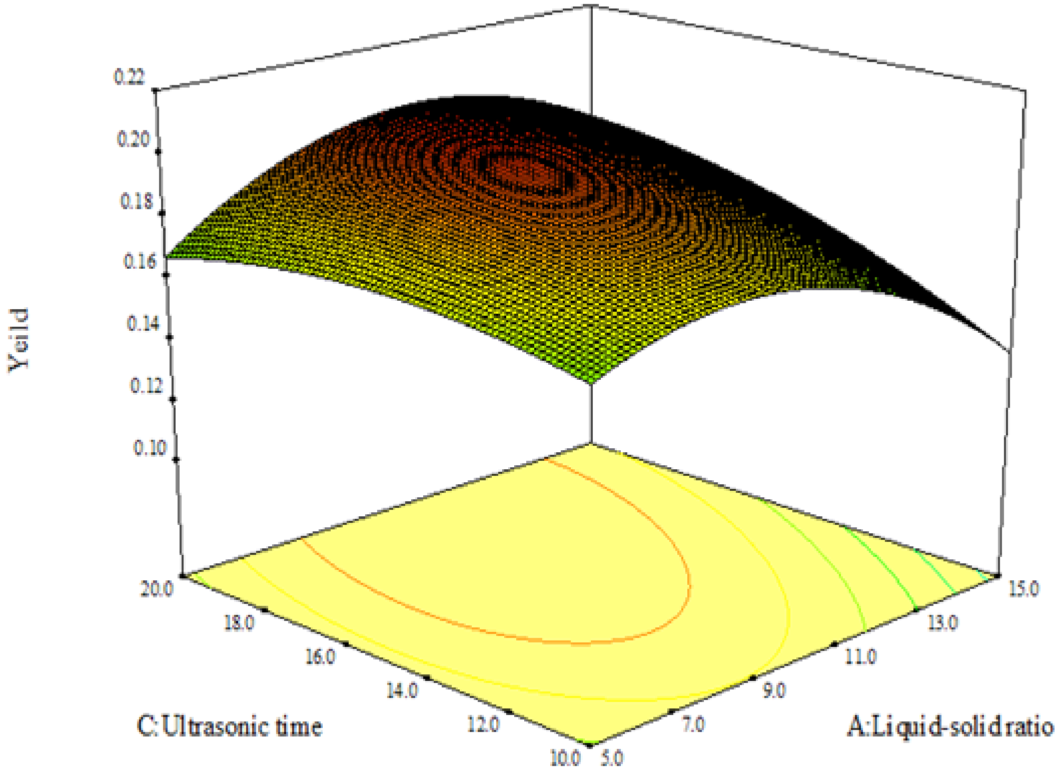


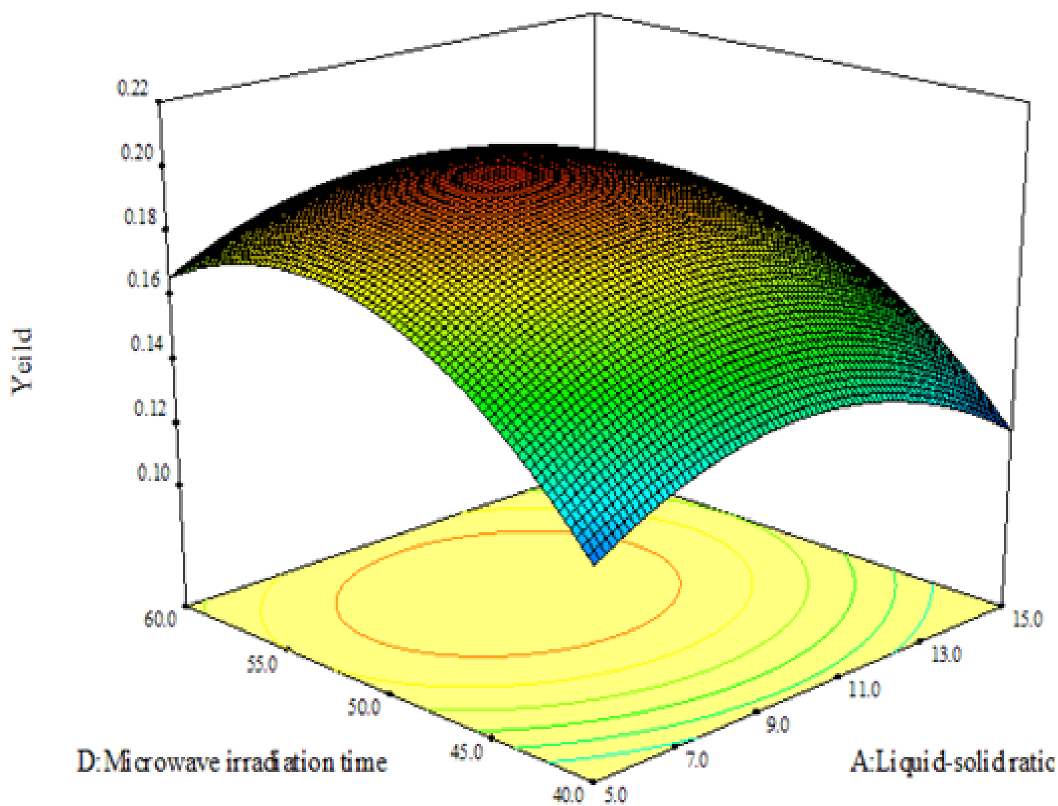
Fig. 1: Perturbation plot showing the effect of process variables



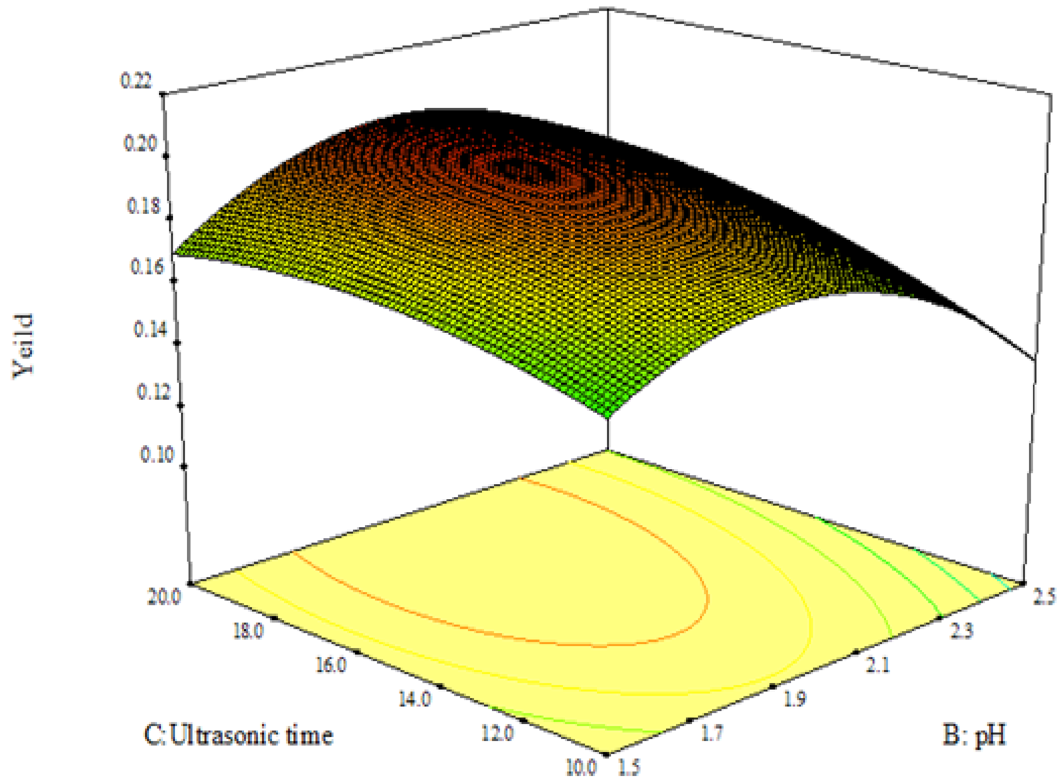
(a)


0.22
0.20
0.18
0.16
0.14
0.12
0.10
Yeild
20.0
18.0
16.0
14.0
12.0
10.0
C:Ultrasonic time
5.0
7.0
9.0
11.0
13.0
15.0
A:Liquid-solid ratio

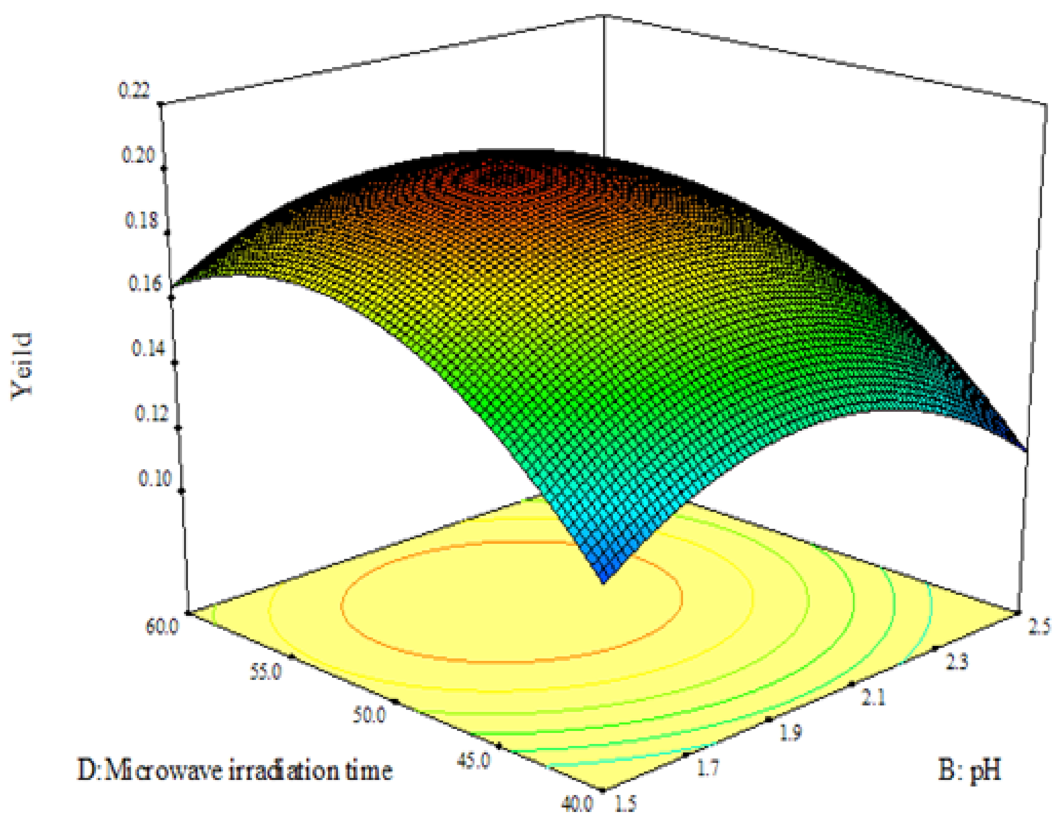
(b)


0.22
0.20
0.18
0.16
0.14
0.12
0.10
Yeild
60.0
55.0
50.0
45.0
40.0
D:Microwave irradiation time
5.0
7.0
9.0
11.0
13.0
15.0
A:Liquid-solid ratic

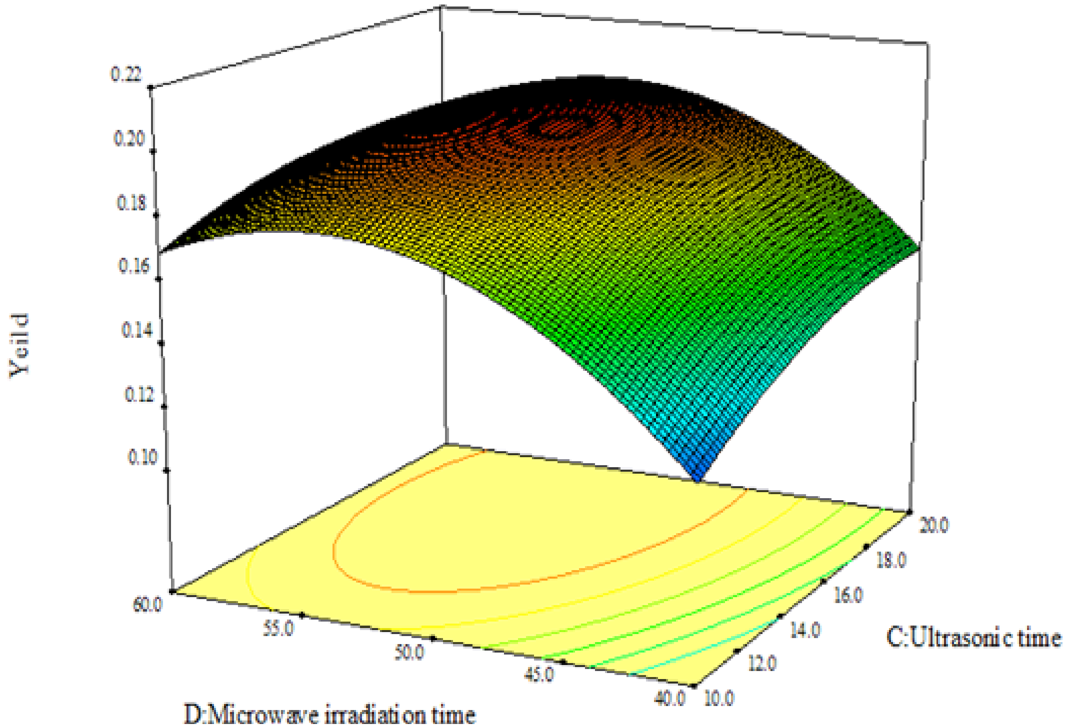
(c)


0.22
0.20
0.18
0.16
0.14
0.12
0.10
Yeild
20.0
18.0
16.0
14.0
12.0
10.0
C: Ultrasonic time
1.5
1.7
1.9
2.1
2.3
2.5
B: pH

(d)


0.22
0.20
0.18
0.16
0.14
0.12
0.10
Yeild
60.0
55.0
50.0
45.0
40.0
D: Microwave irradiation time
1.5
1.7
1.9
2.1
2.3
2.5
B: pH

(e)



(f)

Fig. 2: Surface plots for pectin extraction of jujube waste, (a) figure plot to show the liquid-solid ratio and pH, (b) figure plot to show the liquid: solid ratio and ultrasonic time, (c) figure plot to show the liquid: solid ratio and microwave irradiation time, (d) figure plot to show the pH and ultrasonic time, (e) figure plot to show the pH and microwave irradiation time, (f) figure plot to show the ultrasonic time and microwave irradiation time

was shown that the yield of pectin was first increased rapidly with the increase of solution pH and reached the maximum at 1.97 and then decreased steadily (Fig. 1a to e). The low pH has the ability to contact with the insoluble pectin directly and turn the insoluble pectin into soluble pectin (Wai *et al*., 2009). As a result, the yield of pectin increased with the lower pH and reached the maximum at 1.97. However, when pH was beyond 1.97, the yield of pectin decreased, the reason was that much low solution pH may lead to degradation of pectin chain molecules (Zhang *et al*., 2013), thus affecting the yield of pectin.

Effect of ultrasonic time on the yield of pectin: Time is one of the important factors affecting extraction yield of pectin. Different extraction time would influence the solvent and solid matrix contact. As shown in Fig. 2b, d and f, the extraction yield was increased steadily and reached the maximum at 17.66 min. This phenomenon could be explained that, all the plant cells could be completely cracked because of acoustic cavitation effects caused by the ultrasonic waves in the earlier period of extraction (Minjares-Fuentes *et al*., 2014; Wang *et al*., 2012). So larger contact area between solvent and material was created and the collapse of the bubbles will promote the interpenetration of the solvent into the plant cells to dissolve most of the pectin present in it and increases the extraction yield. However, when the plant cells ruptured such as insoluble substance were also suspended in the extraction liquid, resulting in the lower permeability of the solvent. So the yield was decreased slowly, when the extraction time extended. Thus, the optimum extraction time was 17.66 min.

Effect of irradiation time on the yield of pectin: The reaction time which can affect the yield of pectin is also one of the key factors and it is necessary to select a proper reaction time to assure the maximum of yield of pectin from jujube. To determinate the most appropriate reaction time to the yield of pectin, reaction time 40-60 sec were studied and the results were in Fig. 1c, e and f. As shown in figures, the yield of jujube was significantly increased at the reaction from 40 to 52.73 sec and then decreased steadily. With the reaction time increasing, the thermal accumulation due to the absorption of microwave energy promoted the dissolution process of pectin into solvent (Masmoudi *et al*., 2008) until 52.73 sec and decreased gradually. Too long reaction time may lead to degradation of pectin chain molecules (Kim *et al*., 2004), so the yield of pectin decreased when the reaction beyond 52.73 sec.

Validation of the model: The objective of optimization was to find out the conditions which give the maximum extraction yield of pectin of jujube. The desirability function approach was applied in the optimization process. This numerical optimization technique evaluates a point that maximizes the desirability function. The optimum extraction conditions and the maximum yield of pectin were obtained desirability function approach was liquid solid ratio of 10.03, pH of 1.97, ultrasonic time 17.66 and microwave irradiation time of 52.73 sec and the maximum yield of pectin was 2.02% with a desirability value of 0.976. The suitability of the optimized conditions for predicting the optimum response values was tested experimentally using the selected optimal conditions. Triplicate experiments were performed under the optimized conditions and the mean values (1.95±0.06 for PY) obtained from real experiments, which agree well with the expected value of 2.02%, demonstrating that the optimized conditions agree well with the real experiments.

CONCLUSION

In this present study, the extraction conditions for enhanced extraction of pectin by ultrasound and microwave process were optimized with a four factor three level Box-Behnken response surface design coupled with desirability function methodology. The results showed that, all the process variables had significant effect on the extraction of pectin and a high correlated quadratic polynomial mathematical model was developed. The optimal conditions were determined to be: the liquid-solid ratio of 10.03 mL/g, pH of 1.97, ultrasonic time of 17.66 sec and the irradiation time of 52.73 sec respectively. Under the optimal conditions, the experimental values (1.95±0.06%) agreed with the predicted values (2.02%).

ACKNOWLEDGMENT

This study was supported by grants from Public science and technology research funds projects of Chinese date Brandy (Grant number 31171725 and 3137185) from the National Natural Science Foundation of China. The authors declare that they have no competing financial interests.

REFERENCES

Abou-Zeid, A.A.Z., N.A. Baeshin and A.O. Baghlaf, 1991. The formation of oxytetracycline in a date-coat medium. Bioresource Technol., 37(2): 179-184.

Besbes, S., S. Cheikh-Rouhou, C. Blecker, B. Hentati, C. Deroanne and H. Attia, 2006. Voies de valorisation des pulpes de dattes. MHA, 18(52): 3-7.

Faravash, R.M. and F.Z. Ashtiani, 2007. The effect of pH, ethanol volume and acid washing time on the yield of pectin extraction from peach pomace. Int. J. Food Sci. Tech., 42(10): 1177-1187.

Fishman, M.L., H.K. Chau, P.D. Hoagland and A.T. Hotchkiss, 2006. Microwave-assisted extraction of lime pectin. Food Hydrocolloid., 20(8): 1170-1177.

Gupta, B.S. and J.E. Ako, 2005. Application of guar gum as a flocculant aid in food processing and potable water treatment. Eur. Food Res. Technol., 221(6): 746-751.

Ho, Y.C., I. Norli, A.F.M. Alkarkhi and N. Morad, 2010. Characterization of biopolymeric flocculant (pectin) and organic synthetic flocculant (PAM): A comparative study on treatment and optimization in kaolin suspension. Bioresource Technol., 101(4): 1166-1174.

Kim, W.C., D.Y. Lee, C.H. Lee and C.W. Kim, 2004. Optimization of narirutin extraction during washing step of the pectin production from citrus peels. J. Food Eng., 63(2): 191-197.

Liu, Y., J. Shi and T.A.G. Langrish, 2006. Water-based extraction of pectin from flavedo and albedo of orange peels. Chem. Eng. J., 120(3): 203-209.

Liu, L.S., M.L. Fishman, K.B. Hicks and M. Kende, 2005. Interaction of various pectin formulations with porcine colonic tissues. Biomaterials, 26(29): 5907-5916.

Masmoudi, M., S. Besbes, M. Chaabouni, C. Robert, M. Paquot, C. Blecker and H. Attia, 2008. Optimization of pectin extraction from lemon by-product with acidified date juice using response surface methodology. Carbohyd. Polym., 74(2): 185-192.

Minjares-Fuentes, R., A. Femenia, M.C. Garau, J.A. Meza-Velázquez, S. Simal and C. Rosselló, 2014. Ultrasound-assisted extraction of pectins from grape pomace using citric acid: A response surface methodology approach. Carbohyd. Polym., 106: 179-189.

Ni, M.L. and Q.X. Zeng, 2010. Study on the extraction technology of oat polyphenol by response surface methodology [J]. Sci. Technol. Food Ind., 4: 77.

Prakash Maran, J., V. Mekala and S. Manikandan, 2013a. Modeling and optimization of ultrasound-assisted extraction of polysaccharide from Cucurbita moschata. Carbohyd. Polym., 92(2): 2018-2026.

Prakash Maran, J., V. Sivakumar, K. Thirugnanasambandham and R. Sridhar, 2013b. Optimization of microwave assisted extraction of pectin from orange peel. Carbohyd. Polym., 97(2): 703-709.

Prakash Maran, J., V. Sivakumar, K. Thirugnanasambandham and R. Sridhar, 2014. Microwave assisted extraction of pectin from waste citrullus lanatus fruit rinds. Carbohyd. Polym., 101: 786-791.

Rodriguez-Jasso, R.M., S.I. Mussatto, L. Pastrana, C.N. Aguilar and J.A. Teixeira, 2011. Microwave-assisted extraction of sulfated polysaccharides (fucoidan) from brown seaweed. Carbohyd. Polym., 86(3): 1137-1144.

Sahari, M.A., A. Akbarian and M.M. Hamedi, 2003. Effect of variety and acid washing method on extraction yield and quality of sunflower head pectin. Food Chem., 83(1): 43-47.

Seixas, F.L., D.L. Fukuda, F.R.B. Turbiani, P.S. Garcia, C.L. Petkowicz, S. Jagadevan and M.L. Gimenes, 2014. Extraction of pectin from passion fruit peel (*Passiflora edulisf*. flavicarpa) by microwave-induced heating. Food Hydrocolloid., 38: 186-192.

Urias-Orona, V., A. Rascón-Chu, J. Lizardi-Mendoza, E. Carvajal-Millán, A.A. Gardea and B. Ramírez-Wong, 2010. A novel pectin material: extraction, characterization and gelling properties. Int. J. Mol. Sci., 11(10): 3686-3695.

Wai, W.W., A.F.M. Alkarkhi and A.M. Easa, 2009. Optimization of pectin extraction from durian rind (*Durio zibethinus*) using response surface methodology. J. Food Sci., 74(8): C637-C641.

Wang, X.S., Y.F. Wu, S.L. Dai, R. Chen and Y. Shao, 2012. Ultrasound-assisted extraction of geniposide from *Gardenia jasminoides*. Ultrason. Sonochem., 19(6): 1155-1159.

Wang, S.J., F. Chen, J.H. Wu, Z.F. Wang, X.J. Liao and X.S. Hu, 2007. Optimization of pectin extraction assisted by microwave from apple pomace using response surface methodology. J. Food Eng., 78(2): 693-700.

Westereng, B., T.E. Michaelsen, A.B. Samuelsen and S.H. Knutsen, 2008. Effects of extraction conditions on the chemical structure and biological activity of white cabbage pectin. Carbohyd. Polym., 72(1): 32-42.

Zhang, Y.Y., T.H. Mu and M. Zhang, 2013. Optimisation of acid extraction of pectin from sweet potato residues by response surface methodology and its antiproliferation effect on cancer cells. Int. J. Food Sci. Tech., 48(4): 778-785.