Carbohydrate Polymers 112 (2014) 622–626

Contents lists available at ScienceDirect

Carbohydrate Polymers

journal homepage: www.elsevier.com/locate/carbpol

Short communication

Process optimization and analysis of microwave assisted extraction of pectin from dragon fruit peel

K. Thirugnanasambandham, V. Sivakumar*, J. Prakash Maran

Department of Food Technology, Kongu Engineering College, Perundurai, Erode 638052, TN, India

ARTICLE INFO

Article history:
Received 16 April 2014
Received in revised form 23 May 2014
Accepted 11 June 2014
Available online 27 June 2014

Keywords:
Pectin
Dragon fruit
Microwave extraction
Box–Behnken design
Optimization

ABSTRACT

Microwave assisted extraction (MAE) technique was employed for the extraction of pectin from dragon fruit peel. The extracting parameters were optimized by using four-variable-three-level Box–Behnken design (BBD) coupled with response surface methodology (RSM). RSM analysis indicated good correspondence between experimental and predicted values. 3D response surface plots were used to study the interactive effects of process variables on extraction of pectin. The optimum extraction conditions for the maximum yield of pectin were power of 400 W, temperature of 45 °C, extracting time of 20 min and solid–liquid ratio of 24 g/mL. Under these conditions, 7.5% of pectin was extracted.

© 2014 Elsevier Ltd. All rights reserved.

1. Introduction

Pectin is defined as the methylated ester of polygalacturonic acid that contains 1,4-linked α-D-galacturonic acid residues and these polysaccharides consist of more than 1000 chains of galacturonic acid units (Levigne, Ralet, & Thibault, 2002). It is commonly originate from the cell walls and middle lamellae of higher plants, but is most concentrated in citrus fruits (oranges, lemons, grapefruits) and apples. Currently, commercial pectin's are almost completely derived from citrus peel or apple pomace, both by-products from juice manufacturing. Pectin is widely used in the food industry as a thickener, emulsifier, texturizer and stabilizer and also it is added in jams and jellies as a gelling agent. It has also been used as a fat substitute in spreads, ice-cream and salad dressings. In terms of nutrition, pectin has been shown to lower blood cholesterol levels and low-density lipoprotein cholesterol fractions, which is beneficial for human health (Liu, Shi, & Langrish, 2006). Last few decades, pectin can be obtained from many sources with a variation in the percentage yield. Dragon fruit (*Hylocereus polyrhizus*) or red pitaya is one of the tropical fruits in India that belongs to the cactus family, Cactaceae. The fruits contain high amounts of vitamin C and water-soluble fiber (Ruzainah Ali, Ahmad, Nor Zaini, & Vasudevan, 2009). The average production of dragon fruit in 2014 was 1134 ha with a approximately total production of 3835 tons (Cheah & Wan, 2008). The peels are mostly waste resources resulting from the dragon fruit juice processing industry and are normally discarded in roadsides. These activities cause an environmental problem, thus, in addition to being fed to animals, the peels can be used in the production of pectin, which would then increase the resource recovery as well as reduce the ecological degradation for the dragon fruit juice processing industry.

Last few decades, various extraction techniques were used to extract pectin from different kind of plant materials. Among various kinds of extraction techniques, the efficiency of microwave irradiation is significantly higher, when compared to the conventional extraction methods. When microwave irradiation is used to extract the pectin, two principal mechanisms are simultaneously functioning. One of these is a rapid increase in temperature, which reduces emulsion viscosity and breaks the outer film of sample, thus improve the extraction yield. The other is molecular rotation, which neutralizes the Zeta potential. This phenomenon rearrange the electrical charges surrounding the molecules, resulting in enhanced movement of ions which increase the efficiency of extraction process. However, best of our knowledge MAE is not employed for the extraction of pectin from dragon fruit. Hence, the objectives of this study were to estimate the influence of various MAE extraction conditions on the yield of dragon fruit pectin. Moreover, Response

* Corresponding author. Tel.: +91 4294 226606; fax: +91 4294 220087.
E-mail addresses: drvsivakumar@yahoo.com, thirusambath5@gmail.com (V. Sivakumar).

http://dx.doi.org/10.1016/j.carbpol.2014.06.044
0144-8617/© 2014 Elsevier Ltd. All rights reserved.

Table 1
Process variables and their ranges.

	Level		
	−1	0	1
A	150	350	550
B	35	45	55
C	5	15	25
D	10	20	30

surface methodology (RSM) coupled with Box–Behnken response surface design (BBD) was used to optimize and model the present extraction process.

2. Materials and methods

2.1. Materials and chemicals

The dragon fruit used were collected from Pungamuthur, Udumalpet, TamilNadu and the peels of the fruits were removed and chopped into 1 cm^2 pieces using a stainless steel knife. Then, these pieces were dried in room temperature and were ground in a laboratory dry blender. Finally, dried peels were treated four times with isopropanol (85 vol.%) at 70 °C for 20 min and it was dried, crushed and screened to obtain a fine powder. All the chemicals and reagents used were of analytical grade and were purchased from Sigma–Aldrich, Chennai.

2.2. Extraction procedure

MAE of pectin was performed in an ordinary household microwave oven (LG, MH-3948 WB) at working frequency of 2450 MHz with adjustable microwave power. About 5 g of dried dragon fruit peel powder was weighed and placed into a 1000 ml Pyrex beaker. The beaker was placed in the middle of the oven over a rotating dish and was exposed to microwave radiation at different powers for the selected temperature and solid–liquid ratio. After microwave heating, the mixture in the Pyrex beaker was allowed to cool down and filtered using filter paper (Whatman no-1). The filtered extract was centrifuged and the supernatant was precipitated with an equal volume of 95% (v/v) ethanol. Each of the extraction conditions was carried out in triplicates.

2.3. Analytical method

The extraction yield of pectin was calculated as follows (Prakash Maran, Sivakumar, Thirugnanasambandham, & Sridhar, 2013b),

$$\text{Pectin yield}\,(\%) = \left(\frac{m_0}{m}\right) \times 100 \tag{1}$$

where m_0 (g) is the weight of dried pectin and m (g) is the weight of dried dragon fruit peel powder.

2.4. RSM design

In the present study, RSM coupled with BBD was employed to investigate the individual and interactive effects of process variables on the extraction process. Power, temperature, extraction time and solid–liquid ratio are selected as independent variables (Table 1), whereas extraction yield of pectin (Y) is selected as response. Results were fitted into the empirical second order polynomial model, as shown in the following equation (Thirugnanasambandham, Sivakumar, & Prakash Maran, 2014a)

$$Y = \beta_0 + \sum_{j=1}^{k}\beta_j X_j + \sum_{j=1}^{k}\beta_{jj} X_j^2 + \sum_{i}\sum_{<j=2}^{k}\beta_{ij} X_i X_j + e_i \tag{2}$$

where Y is the response; X_i and X_j are variables (i and j range from 1 to k); β_0 is the model intercept coefficient; β_j, β_{jj} and β_{ij} are interaction coefficients of linear, quadratic and the second-order terms, respectively; k is the number of independent parameters ($k = 4$ in this study); and e_i is the error.

Table 2
BBD experimental design with results.

S. No.	A	B	C	D	Y
1	150	45	15	10	5.02
2	350	45	25	10	5.62
3	350	35	5	20	4.57
4	150	45	5	20	4.73
5	550	45	5	20	4.63
6	350	45	15	20	7.42
7	350	55	15	30	6.16
8	350	45	5	10	4.73
9	550	45	25	20	7.02
10	550	45	15	30	6.42
11	550	55	15	20	5.51
12	350	35	15	10	5.99
13	350	45	15	20	7.42
14	350	45	15	20	7.42
15	350	45	5	30	5.29
16	350	35	25	20	5.18
17	150	45	15	30	5.99
18	350	45	25	30	6.75
19	350	45	15	20	7.42
20	350	45	15	20	7.42
21	550	45	15	10	6.29
22	150	55	15	20	4.52
23	350	55	5	20	4.02
24	550	35	15	20	5.73
25	150	35	15	20	4.9
26	150	45	25	20	5.26
27	350	55	25	20	5.51
28	350	55	15	10	4.72
29	350	35	15	30	4.92

3. Results and discussions

3.1. Mathematical modeling

In the present study, extraction yield of pectin was investigated according to BBD (29 batch experiments) and its results are shown in Table 2. Then, BBD experimental data are investigated using multi regression analysis namely the sequential model sum of squares (Table 3). From the results, second order polynomial equation was selected to represent the MAE extraction process, due to higher F-value with low p-value. The equation with coded factors was given below

$$\begin{aligned} Y = {} & 7.42 + 0.43A - 0.071B + 0.61C + 0.26D + 0.040AB \\ & + 0.47AC - 0.21AD + 0.22BC + 0.63BD + 0.14CD - 0.85A^2 \\ & - 1.39B^2 - 1.19C^2 - 0.62D^2 \end{aligned} \tag{3}$$

where Y is extraction yield of pectin (%); A, B, C and D are power, temperature, extraction time and solid–liquid ratio respectively. ANOVA results of the quadratic model in Table 4 indicated that the model equation derived by RSM could adequately be used to describe the extraction process under a wide range of operating conditions. As can be seen in Table 4, F-value was >27 and implied that the quadratic model was significant. Moreover, each term in the model was also tested for significance. A p-value less than 0.05

Table 3
Sequential model sum of squares for extraction yield of pectin.

Source	Sum of squares	df	Mean square	F value	Prob > F	Remarks
Sequential model sum of squares for extraction yield of pectin (%)						
Mean	956.86	1.00	956.86			
Linear	7.65	4.00	1.91	1.99	0.1282	
2FI	2.90	6.00	0.48	0.43	0.8488	
Quadratic	19.81	4.00	4.95	189.26	<0.0001	Suggested
Cubic	0.35	8.00	0.04	19.12	0.0010	Alised
Residual	0.01	6.00	0.00			
Total	987.59	29.00	34.05			

Table 4
ANOVA results for extraction yield of pectin.

Source	Sum of squares	df	Mean square	F value	P-value
Model	30.36	14.0000	2.16868	82.8777	<0.0001
A	2.24	1.0000	2.23603	85.4516	<0.0001
B	0.06	1.0000	0.06021	2.3009	0.1516
C	4.53	1.0000	4.52641	172.98	<0.0001
D	0.83	1.0000	0.83213	31.8006	<0.0001
AB	0.01	1.0000	0.0064	0.24458	0.6286
AC	0.86	1.0000	0.8649	33.0528	<0.0001
AD	0.18	1.0000	0.1764	6.74125	0.0211
BC	0.19	1.0000	0.1936	7.39856	0.0166
BD	1.58	1.0000	1.57503	60.1907	<0.0001
CD	0.08	1.00	0.08	3.10	0.0999
A^2	4.71	1.00	4.71	180.15	<0.0001
B^2	12.51	1	12.51	478.079	<0.0001
C^2	9.20482	1	9.20482	351.769	<0.0001
D^2	2.47334	1	2.47334	94.5203	<0.0001
CV			2.82		
AP			29.95		

implies that the corresponding model term is significant. Moreover, individual and interactive model terms also shows the significance of the developed model. The obtained CV of this study is not greater than 10% and shows the reproducible nature. In the present study, adequate precision value was obtained as >4, demonstrate a significant and intense correlation between the observed and predicted values. The actual and predicted pectin yields are plotted in Fig. 1. Actual values are data for each specific run from Table 2, and predicted values are produced by the model, Eq. (3). The data points on this plot lie reasonably close to the straight line and indicate that an adequate agreement between real data and the data obtained from the model (Prakash Maran, Sivakumar, Thirugnanasambandham, & Sridhar, 2013a).

3.2. Effect of extraction conditions on the pectin yield

In order to study the interactive effect of the process variables on the response, response surface contour plots are plotted from the developed mathematical model (Eq. (3)) and it is shown in Fig. 2. Power is one of the crucial parameter which influence the

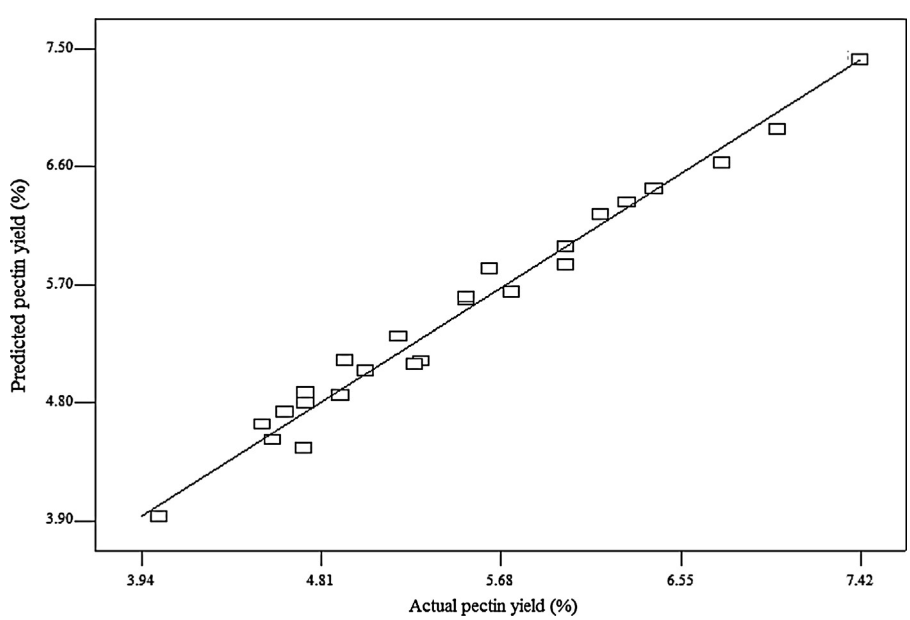

Fig. 1. Predicted versus actual plot for pectin yield.

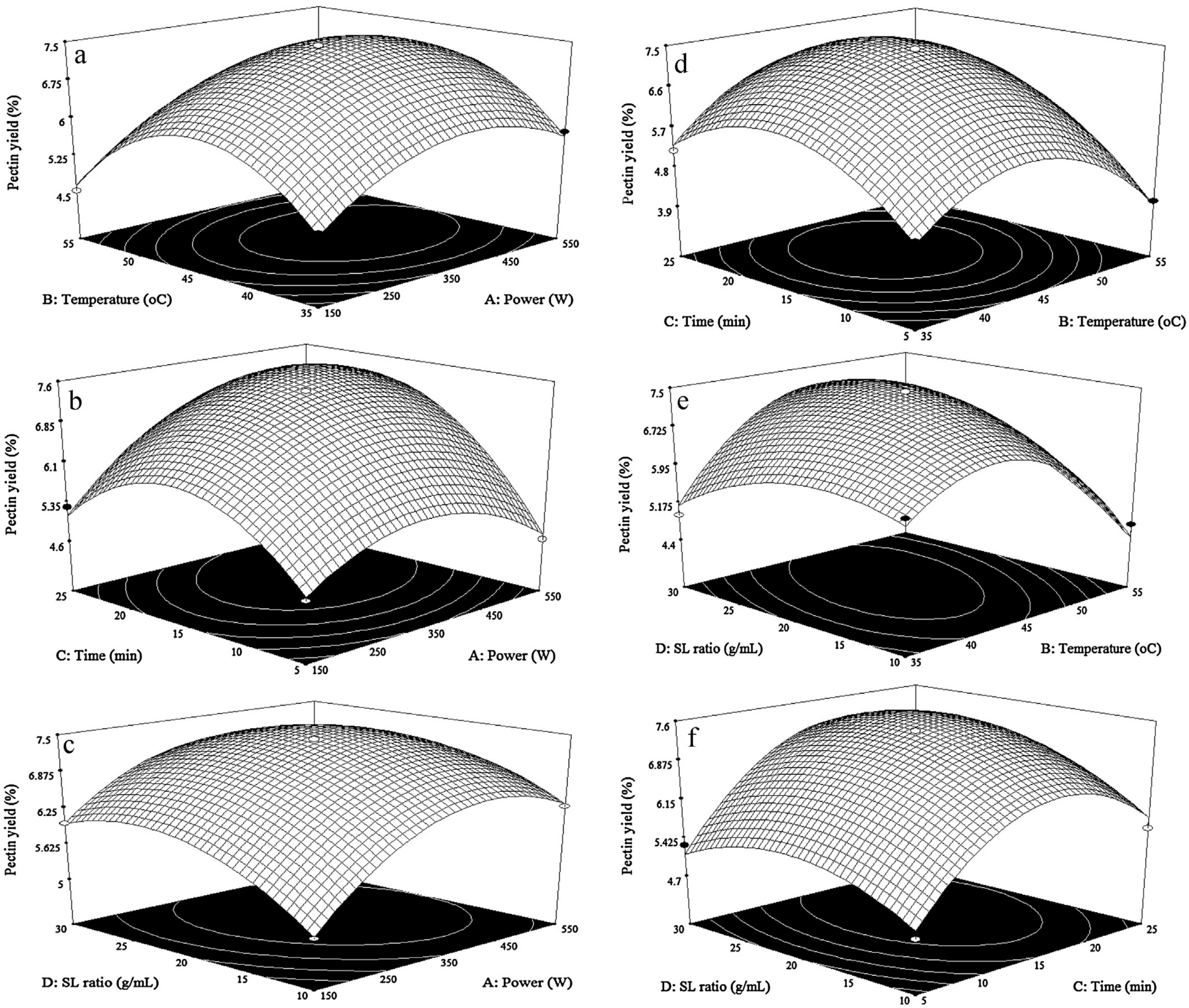

Fig. 2. Response surface plots representing the effect of process variables on extraction yield.

extraction of pectin from dragon fruit peel. In order to investigate the effect of power on pectin yield, experiments were carried out in various power and the results are shown in Fig. 2a–c. From the results, it is observed that, the pectin yield is increased linearly with increasing power up to 400 W (Prakash Maran, Manikandan, & Mekala, 2013). However, thereafter, power shows the negative impact on extraction of pectin. Temperature is the key parameter which influence the present extraction process significantly. So that, experiments were carried out to study the effect of temperature on the pectin yield. From the results, it is observed that, the pectin yield is increased linearly with increasing temperature from 35 to 45 °C (Fig. 2a, d and e). However, temperature beyond 45 °C resulted in lower pectin yield (Lee, Chung, & Lee, 2008).

Time is one of the important process variable for extraction of pectin and it is associated with the mass transfer effect. To examine its effect on extraction process, experiments were carried out in various extraction time and the results are shown in Fig. 2b, d and f. From the results, it is found that, the pectin yield is increased rapidly with increasing the time up to 20 min. Beyond 20 min shows the negligible pectin yield (Happi, Ronkart, Robert, Wathelet, & Paquot, 2008). SL ratio is a one of the crucial parameter which influences the extraction process appreciably. In order to investigate the effect of SL ratio, the experiments were carried out at various SL ratio and the results are illustrated in Fig. 2c, e and f, which shows that pectin yield increased with increasing SL ratio up to 25 g/mL. There after there is a drastic decrease in percentage yield of pectin from dragon fruit peel. Perturbation plot for extraction yield of pectin is shown in Fig. 3 and it shows the effect of process variables on the extraction process deviation from reference point (Gnanasambandam & Proctor, 1999).

3.3. *Multi response optimization*

According to BBD results, optimal extraction conditions to obtain the maximum extraction yield of pectin from dragon fruit peel is determined by Derringer's desired function methodology (Thirugnanasambandham, Sivakumar, & Prakash Maran, 2014b) as follows: power of 400 W, temperature of 45 °C, extracting time of 20 min and solid–liquid ratio of 24 g/mL. Under this conditions, 7.5% of pectin was extracted with a desirability value of 0.926 (Thirugnanasambandham, Sivakumar, Prakash Maran, & Kandasmay, 2014). The suitability of the optimized conditions for predicting the pectin yield value was tested experimentally using the selected optimal conditions. Triplicate experiments were

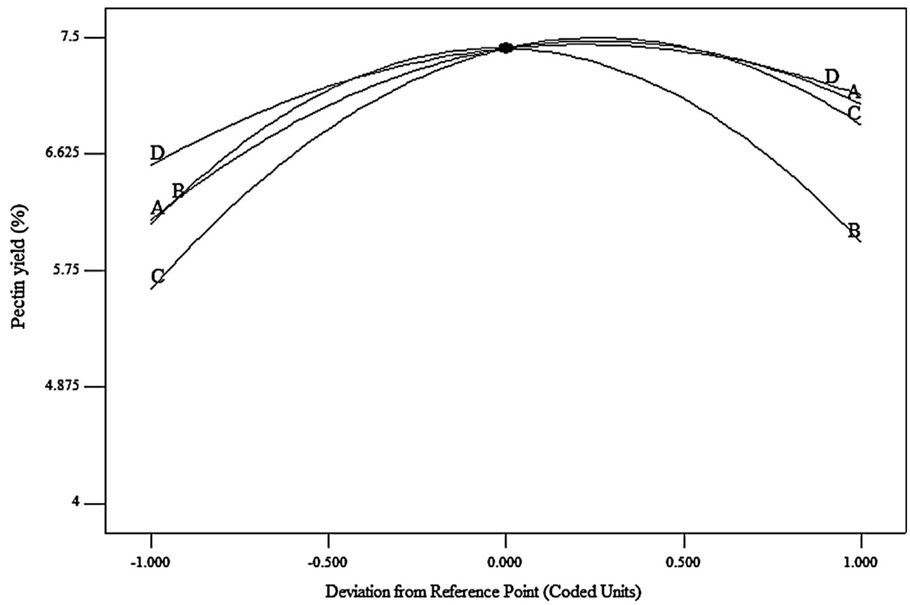

Fig. 3. Perturbation plot for extraction yield of pectin.

performed under the optimized conditions and the mean values (7.44%) obtained from real experiments, demonstrated the validation of the optimized conditions (Prakash Maran, Sivakumar, et al., 2013a).

4. Conclusions

In the present study, extraction of pectin from dragon fruit peel is investigated under different extraction conditions such as power, temperature, extracting time and solid–liquid ratio. Four factors at three levels BBD coupled with RSM was used to develop the second order polynomial model. Maximum extraction yield of pectin from dragon fruit peel is found to be as follows: power of 400 W, temperature of 45 °C, extraction time of 20 min and solid–liquid ratio of 24 g/mL. Under these optimal conditions, 7.5% of pectin was extracted. FTIR confirms the presence of various functional groups in extracted pectin. Results exhibited that, MAE is found to be a suitable technique to extract the pectin from dragon fruit peel.

References

Cheah, L. S., & Wan, M. Z. (2008). *Status of pitaya cultivation in Malaysia*. Putrajaya: D.O. Agriculture.

Gnanasambandam, R., & Proctor, A. (1999). Preparation of soy hull pectin. *Food Chemistry*, *65*(4), 461–467.

Happi, E. T., Ronkart, S. N., Robert, C., Wathelet, B., & Paquot, M. (2008). Characterisation of pectins extracted from banana peels (*Musa* AAA) under different conditions using an experimental design. *Food Chemistry*, *108*, 463–471.

Lee, J.-S., Chung, D., & Lee, H. G. (2008). Optimization of calcium pectinate gel beads for sustained-release of catechin using response surface methodology. *International Journal of Biological Macromolecules*, *42*, 340–347.

Levigne, S., Ralet, M.-C., & Thibault, J.-F. (2002). Characterisation of pectins extracted from fresh sugar beet under different conditions using an experimental design. *Carbohydrate Polymers*, *49*, 145–153.

Liu, Y., Shi, J., & Langrish, T. A. G. (2006). Water-based extraction of pectin from flavedo and albedo of orange peels. *Chemical Engineering Journal*, *120*, 203–209.

Prakash Maran, J., Manikandan, S., & Mekala, V. (2013). Modeling and optimization of betalain extraction from *Opuntia ficus-indica* using Box–Behnken design with desirability function. *Industrial Crops and Products*, *49*, 304–311.

Prakash Maran, J., Sivakumar, V., Thirugnanasambandham, K., & Sridhar, R. (2013b). Optimization of microwave assisted extraction of pectin from orange peel. *Carbohydrate Polymers*, *97*, 703–709.

Prakash Maran, J., Sivakumar, V., Thirugnanasambandham, K., & Sridhar, R. (2013c). Response surface modeling and analysis of barrier and optical properties of maize starch edible films. *International Journal of Biological Macromolecules*, *60*, 412–421.

Ruzainah Ali, J., Ahmad, Ridhwan Bin A. R., Nor Zaini, C. M., & Vasudevan, R. (2009). Proximate analysis of dragon fruit (*Hylecereus polyhizus*). *American Journal of Applied Sciences*, *6*, 1341–1346.

Thirugnanasambandham, K., Sivakumar, V., & Prakash Maran, J. (2014). Optimization of electrocoagulation process to treat grey wastewater in batch mode using response surface methodology. *Journal of Environmental Health Science and Engineering*, 12. http://dx.doi.org/10.1186/2052-336X-12-29

Thirugnanasambandham, K., Sivakumar, V., Prakash Maran, J., & Kandasmay, S. (2014). Chitosan based grey wastewater treatment—A statistical design approach. *Carbohydrate Polymers*, *99*, 593–600.

Thirugnanasambandham, K., Sivakumar, V., & Prakash Maran, J. (2014). Efficiency of electrocoagulation method to treat chicken processing industry wastewater—Modeling and optimization. *Journal of the Taiwan Institute of Chemical Engineers*, http://dx.doi.org/10.1016/j.jtice.2014.04.011