Original article

Optimisation of pectin acid extraction from passion fruit peel (*Passiflora edulis* flavicarpa) using response surface methodology

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(Received 12 March 2008; Accepted in revised form 1 April 2008)

Summary Pectin was extracted from passion fruit peel using three different acids (citric, hydrochloric or nitric) at different temperatures (40–90 °C), pH (1.2–2.6) and extraction times (10–90 min), with and without skins using a 2^4 factorial design. Temperature, pH and extraction time had highly significant effects on the pectin yield. A central composite design with face centring was used to optimise the extraction process conditions for citric acid without skins. Pectin yields varied from 10% to 70%. The optimal conditions for maximisation of pectin yield were the use of citric acid at 80 °C and pH 1 with an extraction time of 10 min considering model extrapolation.

Keywords Central composite design, extraction yield, passion fruit, pectin, response surface methodology.

Introduction

Pectin consists of a linear backbone of randomly connected (1 → 4)-linked α-D-galacturonyl units partially esterified with methanol. The galacturonyl units are occasionally interrupted by (1 → 2)-linked α-L-rhamnopyranosyl residues. The homogalacturonan sections are called 'smooth' and the rhamnogalacturonic regions are called 'hairy'. Neutral sugars are also present as side chains in different amounts depending on the pectin source and on the extraction method used (Kjoniksen *et al*., 2005). Pectins extracted from several plant by-products are widely used in the food industry as gelling agents (May, 1990; Pilnik & Voragen, 1992). Depending on their degree of methoxylation (DM), pectins are referred to as high methoxy pectins (HMP) (DM ≥ 560) or low methoxy pectins (LMP) (DM < 50). HMP forms gels in an acidic medium (pH 2.0–3.5) if sucrose is present at a concentration higher than 55 wt%. LMP can gel over a larger pH range (2.0–6.0) in the presence of a divalent ion, such as calcium. In this case, the presence of sucrose is not necessary for forming the gel (Mishra *et al*., 2001; Neirynck *et al*., 2004; Kjoniksen *et al*., 2005). These applications account for the substantial consumption of pectin worldwide.

An extraction process is the most important operation to obtain pectin from vegetal tissue. Pectin extraction is a multiple-stage physical–chemical process in which hydrolysis and extraction of pectin macromolecules from plant tissue and their solubilisation take place under the influence of different factors, mainly temperature, pH and time (Kertesz, 1951).

Pectin extraction has been studied by several authors. El-Nawawi & Shehata (1987) investigated the factors affecting the extraction of pectin from orange peel where the maximum yield was obtained using hydrochloric acid (90 °C, pH 1.7 and 120 min). Pagán & Ibarz (1999) studied the extraction and the rheological properties of pectin from peach pomace, where the maximum yield was obtained using 70% nitric acid, 80 °C, pH 1.2 and 60 min. Virk & Sogi (2004) studied pectin extraction and characterisation from apple peel waste and revealed that citric acid was more effective than hydrochloric acid. Rehmann *et al*. (2004) extracted pectin from mango peels with sulfuric acid, and the maximum yield was obtained at 80 °C and pH 2.5 with an extraction time of 120 min. Schemin *et al*. (2005) carried out a practical follow-up to pectin extraction from apple pomace and observed that the pectin yield was higher with 6.2 g per 100 mL of citric acid and a reaction time around 150 minutes. Faravash & Ashtiani (2007) determined

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doi:10.1111/j.1365-2621.2008.01753.x

the effects of extraction time and pH variation on the yield of pectin isolation from pomace peento peaches, and the maximum yield of pectin was obtained at initial pH 2.5, EV of 1.5 and acid washing time of 120 min.

There is only one factory in Brazil producing citrus pectin, in Limeira, state of São Paulo, but none produces passion fruit pectin. Brazil however is the major passion fruit producer with an estimated 485 000 tons in 2003. Passion fruit peel is a by-product of the juice factories, and currently, it is either used for animal feed or is disposed of as industrial waste.

Considering that in pectin manufacture by the food industry, the whole peel is extracted, the purpose of this study was to investigate the extractability of pectin from passion fruit waste using the following variables: pH, temperature and extraction time, with and without skins, with three different kinds of acids, i.e. citric, hydrochloric or nitric. A full 2^4 experimental design 2^4 was carried out for the variable screening, and a CCD was used for the optimisation of pectin yield.

Materials and methods

Materials

Passion fruits at the same ripeness stage were obtained from the CEASA fruit farm during the months of January to May of 2005.

The raw material was prepared for the experiments in the following way: all the fruits were first washed and the pulp was then separated from the fruit flesh. The peel was divided into two portions. From one lot, the skin was removed, while in the other one, it was not. Both lots of peel were dried in an air-forced oven at 55 °C until constant weight. The dried passion fruit peels were then milled to a dry 60-sieve-size powder. The ground powders were packaged in polyethylene bags and stored at refrigerator temperature until required.

A standardised commercial citrus LMP (Genu® pectin, DM = 34%) (Hercules, Copenhagen, Denmark) was used as reference. All the chemical reagents used were of analytical grade.

Pectin extraction

The extraction procedure was based on that of Kratchanova *et al*. (2004), considering several variables. A dry mass (5 g) was subjected to extraction by adding 250 mL of water. The pH was adjusted to 1.2–2.6 with 0.5 M HCl, 0.5 M HNO_3 or citric acid. The mixture was then heated to 45, 65 or 90 °C and the extraction was carried out with continuous stirring for 10, 45 or 90 min. The hot acid extract was filtered through the ordinary screen with 1-mm mesh size equipped with two-layer cheesecloth, and the filtrate was cooled down to 4 °C. The filtrate was coagulated using an equal volume of 96% ethanol and left for 1 h. The coagulated pectin was separated by filtration, washed once with 70% acidic ethanol (0.5% HCl), then with 70% ethanol to a neutral pH and finally with 96% ethanol. The resulting material was dried overnight at 55 °C in an air-forced oven.

The pectin yield is calculated using eqn 1:

$$y_{\text{pec}}(\%) = 100\left(\frac{P}{B_i}\right) \tag{1}$$

where y_{pec} is the extracted pectin yield in per cent (%), P is the amount of extracted pectin in g and B_i is the initial amount of ground passion fruit peel (5 g).

Experimental design

Full factorial design

A two-level full factorial design, 2^4, was carried out with an aim to investigate the effects of the variables on the response y_{pec}: the variables investigated were pH, temperature (T), extraction time (ET) and presence of skin (PS) in three different acids. Table 1 illustrates the factors under investigation and the levels of each factor used in the experimental design. The levels were selected based on results obtained in preliminary studies (Kliemann, 2005). The experiments were performed in random order and in duplicate.

Central composite design

In order to describe the nature of the surface response in the experimental region, a central composite design (CCD) was applied. The CCD was described by Box & Wilson, 1951 as an evolution of the 3^3 designs that required many experiments for only a few factors, even for fractional designs. Advantages like rotatability, orthogonal blocking and the requirement of fewer assays were obtained owing to the presence of the following parts in the design (Myers & Montgomery, 2002): (i) a two-level full factorial design; (ii) central point experiments; (iii) axial point experiments that are situated at the centre of the axis system with distances equal to $\pm\alpha$ from the origin, which composes the star region of the design, where $\alpha = \sqrt[4]{2^k}$.

Fitting model

If all variables are assumed to be measurable, the response surface can be expressed as follows:

$$y = f(x_1, x_2, x_3, \ldots, x_n) \tag{2}$$

where y is the result from the system, and x_n are the variables of action called factors. The goal is to optimise the response variable y. It is assumed that the independent variables are continuous and controllable by experiments with negligible errors. It is necessary to find a suitable approximation for the true functional

Table 1 Factors coded (in bracket) and decoded levels used in the full experimental design and the mean response obtained using the three different acids

	Actual and coded level of variables*				Average experimental responses†		
Run	pH	*T* (°C)	ET (min)	PS	Citric acid	HCl	HNO_3
8	2.6 (+1)	90 (+1)	90 (+1)	Absent (−1)	41.21	26.02	27.72
14	2.6 (+1)	40 (−1)	90 (+1)	Present (+1)	24.55	14.68	12.52
2	2.6 (+1)	40 (−1)	10 (−1)	Absent (−1)	16.01	16.14	11.18
6	2.6 (+1)	40 (−1)	90 (+1)	Absent (−1)	23.79	14.35	14.76
12	2.6 (+1)	90 (+1)	10 (−1)	Present (+1)	36.76	15.30	16.36
4	2.6 (+1)	90 (+1)	10 (−1)	Absent (−1)	37.25	17.81	20.47
9	1.2 (−1)	40 (−1)	10 (−1)	Present (+1)	34.52	15.08	13.48
13	1.2 (−1)	40 (−1)	90 (+1)	Present (+1)	57.38	13.89	14.72
3	1.2 (−1)	90 (+1)	10 (−1)	Absent (−1)	66.76	24.02	24.52
7	1.2 (−1)	90 (+1)	90 (+1)	Absent (−1)	61.34	24.40	26.68
15	1.2 (−1)	90 (+1)	90 (+1)	Present (+1)	60.09	18.43	22.21
1	1.2 (−1)	40 (−1)	10 (−1)	Absent (−1)	38.28	12.19	26.14
11	1.2 (−1)	90 (+1)	10 (−1)	Present (+1)	60.30	17.90	18.94
10	2.6 (+1)	40 (−1)	10 (−1)	Present (+1)	16.60	11.76	8.94
16	2.6 (+1)	90 (+1)	90 (+1)	Present (+1)	34.88	22.15	24.60
5	1.2 (−1)	40 (−1)	90 (+1)	Absent (−1)	57.13	14.74	21.22

*T, temperature (°C); ET, extraction time (min); PS, presence of skin.
†y_{pec} (%).

relationship between independent variables and the response surface. Usually a second-order model is utilised in response surface methodology (RSM). In general, this model can be written in matrix form eqn 3.

$$\hat{\mathbf{y}} = \mathbf{X}\hat{\mathbf{b}} \tag{3}$$

where $\hat{\mathbf{y}}$ is defined to be a vector of estimated values and $\mathbf{X}$ is a matrix of independent variables. The vectors $\hat{\mathbf{b}}$ and $\mathbf{e}$ consist of coefficients and errors, respectively. The regression vector $\hat{\mathbf{b}}$ can be obtained by the approach expressed in eqn 4 (Box & Draper, 1987; Teófilo & Ferreira, 2006):

$$\hat{\mathbf{b}} = (\mathbf{X}'\mathbf{X})^{-1}\mathbf{X}'\mathbf{y} \tag{4}$$

where $\mathbf{X}'$ is the transpose of the matrix $\mathbf{X}$ and $(\mathbf{X}'\mathbf{X})^{-1}$ is the inverse of the product matrixes $\mathbf{X}'\mathbf{X}$. Equation 4 is well-known as the least squares approach.

The model quality was evaluated by the correlation coefficient (R^2), considering the measured values (y_i) vs. the ones predicted by the model ($\hat{y}_i$'), and evaluation of the residuals plot ($y_i - \hat{y}_i$ vs. y_i). These parameters indicate that if all the data variance around the mean was explained by the regression model. The R^2 value can be interpreted as the proportion of variability around the mean for the dependent variable that can be accounted for by the respective model. A plot of residuals with random distribution indicates that the model is well fitted.

In this work, the standard errors of the effect and the coefficient evaluations were obtained by the mean square residual (MS residual), according to eqn 5, because the pure error presented a very low value owing to the high precision of the yield values obtained experimentally.

$$\text{MS residual} = \frac{\sum_{i=1}^{m}\sum_{j=1}^{r}(y_{ij} - \hat{y}_i)^2}{n - q} \tag{5}$$

where m is the total level number (experimental design points), r is the total replicate number, n - q is the number of degrees of freedom (df) the quadratic residual sum, n is the number of assays and q is the number of calculated parameters (coefficients or effects).

The significance evaluations on the statistical decision were carried out by applying the t test through the P value. An alternative way to evaluate the hypothesis test is comparing the P value of the sample population statistical test with a significance level α. The p value of the sample population statistical test is the lowest significance level needed to reject the null H_0 hypothesis (mean values are equal). In this way, it is necessary to compare the P value with α and, when $P \leq \alpha$, H_0 is rejected; otherwise, H_0 is accepted. Once P is known, all the significance levels can be evaluated allowing that the observed result could be statistically rejected. Specifically, the P value represents the probability of validity of the involved error in the observed result; the

representativeness of the population. Each test was accomplished with their respective df and a significance level (α) of 0.05 (Teófilo & Ferreira, 2006).

The error of the factorial design was obtained in conformity with eqn 6, and the standard errors (SE) of the CCD were acquired according to eqns 7 and 8.

$$\text{Error} = \sqrt{\frac{\text{MS residual}}{n}} \tag{6}$$

$$\mathbf{V}(b) = (\mathbf{X}'\mathbf{X})^{-1} \cdot \text{MS residual} \tag{7}$$

$$\text{SE} = \pm\sqrt{v(b)_{ii}} \quad i = 1,\ 2,\ \ldots,\ k \tag{8}$$

Equation 7 provides the matrix $\mathbf{V}(b)$ (variance–covariance matrix). This matrix is symmetric and its diagonal elements, v_{ii} $i = 1, 2, \ldots, n$, are the variances of the regression parameters given in the same order as they are in the regression equation. The square root of v_{ii} determines the corresponding S.E. values of the calculated coefficients (eqn 8).

All calculations and graphics in this work were performed using electronic worksheets from Microsoft® Excel 2003 in accordance with Teófilo & Ferreira (2006).

Pectin characterisation

Commercial LMP and pectin samples extracted in optimised condition (with highest yield) (CEP) were selected for analysis. Analyses were performed at least in duplicates.

Degree of methoxylation

The DM of pectin samples were determined by the potentiometric titration method of Bochek *et al.* (2001).

Galacturonic acid

The galacturonic acid (GalA) content was determined with a colorimetric method described by Filisetti-Cozzi & Carpita (1991). Samples were dissolved in distilled water (0.5 mg mL^{-1}) under gentle magnetic stirring. To a 400-μL sample (0.5 mg mL^{-1}) in a test tube kept on ice, 40 μL of a 4.0 M sulfamic acid–potassium sulfamate solution (pH 1.6) were added and mixed thoroughly. Analytical grade (96.4%) H_2SO_4 containing 75 mM sodium tetraborate (2.4 mL) was added, and the solution stirred vigorously by vortex mixing. The solution was incubated for 20 min in a boiling water bath. After cooling, 40 μL of 0.15% (w/v) *m*-hydroxydiphenyl in NaOH 0.5% (w/v) was added and the mixture stirred vigorously by vortex mixing. The pink colour develops to completion in about 5 to 10 min, and is stable for about 1 h. Absorbance was read at 525 nm using a standard curve with GalA.

Acetyl value

Acetyl value (AcOH) of pectin samples was determined by the colorimetric method based on hydroxamic acid reaction (Ranganna, 1977). Pectin samples (0.5 g) were dissolved in 0.1 N NaOH solution with stirring and allowed to stand overnight. The contents were diluted to 50 mL with distilled water and an aliquot (20 mL) was placed into the distillation apparatus. Magnesium sulphate–sulfuric acid solution (20 mL) was also transferred to distillation apparatus, distilled, and about 100 mL of distillate was collected. The distillate was titrated with 0.5 N NaOH using phenol red indicator. A blank distillation using 20 mL of the magnesium sulphate–sulfuric acid solution was carried out and the distillate was titrated.

The AcOH was calculated using eqn 9:

$$\text{Acetyl value}(\%) = \frac{\text{N}_{\text{NaOH}} \times \text{mL}_{\text{NaOH}} \times 4.3}{\text{Wt. of sample}} \tag{9}$$

where, N_{NaOH} is normality of NaOH; mL_{NaOH} is the total volume of NaOH required to titrate distillate – total mL required to titrate distillate of blank run; Wt. of sample is the weight of sample in grams, in 20 mL, aliquot.

Results and discussion

Evaluation of the factors affecting pectin yield

Table 1 shows the factors investigated in the 2^4 factorial design, the coded and decoded levels, and the pectin mean response yield of two replicates for the three acid extractors. The factors are abbreviated as *pH*, *T*, ET and PS for pH, temperature, extraction time and presence or not of skins, respectively.

Table 2 shows the effects observed on the studied factors for the response for the three acids, in addition to those caused by the interactions among the factors. The *t* test was accomplished with 21 df.

Citric acid was the best acid for the extraction of pectin. This is in agreement with the results reported by Virk & Sogi (2004) and Schemin *et al.* (2005), who compared the yields of pectin extracted from apple with different acids. Between the two strong acids, it was observed that there was no great difference in the pectin yield, in spite of the effects of nitric acid being slightly larger than those of hydrochloric acid. Even though a low pH is necessary to improve the yield, the strong acid solution could lead to smaller pectin particles owing to partial hydrolysis. Consequently, pectin solubility would increase to the point that no precipitate was formed by

Table 2 Effects and errors obtained from the 2^4 full factorial designs for citric, hydrochloric and nitric acids

	Citric acid				HCl				HNO_3			
	Effect	**Error**	***t***	***P***	**Effect**	**Error**	***t***	***P***	**Effect**	**Error**	***t***	***P***
Mean	41.68*	0.52	80.12	0.000	17.43*	0.31	55.43	0.000	19.03*	0.21	89.71	0.000
pH	−25.60*	1.04	−24.60	0.000	−0.30	0.63	−0.48	0.636	−3.92*	0.42	−9.25	0.000
T	16.29*	1.04	15.66	0.000	6.65*	0.63	10.57	0.000	7.32*	0.42	17.24	0.000
ET	6.74*	1.04	6.48	0.000	2.31*	0.63	3.67	0.001	3.05*	0.42	7.19	0.000
PS	−2.09	1.04	−2.01	0.058	−2.56*	0.63	−4.07	0.001	−5.12*	0.42	−12.06	0.000
pH–*T*	1.00	1.04	0.96	0.349	−0.56	0.63	−0.89	0.382	3.12*	0.42	7.36	0.000
pH–ET	−2.28*	1.04	−2.19	0.040	1.74*	0.63	2.77	0.012	2.61*	0.42	6.16	0.000
pH–PS	0.72	1.04	0.69	0.497	−0.05	0.63	−0.08	0.940	2.19*	0.42	5.16	0.000
T–ET	−7.63*	1.04	−7.33	0.000	1.69*	0.63	2.68	0.014	2.18*	0.42	5.13	0.000
T–PS	−1.54	1.04	−1.48	0.153	−2.06*	0.63	−3.27	0.004	0.80	0.42	1.88	0.075
ET–PS	0.45	1.04	0.43	0.672	−0.03	0.63	−0.05	0.962	1.03*	0.42	2.44	0.024

ET, extraction time (min); PS, presence of skin.
*Significant effects. df = 21, $\alpha = 0.05$.

the addition of alcohol. As noted by Kalapathy & Proctor (2001), this could be the reason why the use of a stronger acid resulted in a lower pectin yield.

The main effect of the skin variable was not significant for citric acid, but was significant and negative for hydrochloric and nitric acids. Consequently, the skin was removed from subsequent experimental studies in this work. The other variables turned out to be meaningful for all acids.

As citric acid was the best for pectin extraction, it was decided to study only this acid when applying RSM.

Optimisation of acid extraction of pectin by central composite design

In order to optimise the pectin extraction from passion fruit, the significant independent variables (*pH*, *T* and ET) were further explored, each at three levels using the CCD with face-centred star points, i.e. $\alpha \pm 1$. This design was chosen because it was not in the authors' interest to investigate other design levels, and it was necessary to build the response surface. Thus, no dislocating or axial points with $\alpha > 1$ were carried out. The experiments were realised on pectin powder prepared without the skin according to Table 1, which is the basis of the factorial portion in the CCD. Therefore, only seven experiments were carried out and included in the earlier set of factorial experiments already done with citric acid (Table 1).

The central and axial experimental points in the face-centred design are shown in Table 3 together with the experimental pectin yields according to CCD. The experiments were carried out in a random order and in duplicate.

The regression coefficients of the response function and the parameters from statistical analysis for citric are given in Table 4, based on the citric acid data from Tables 1 and 3.

Table 3 The coded (in bracket) and decoded levels for the central composite design and response pectin yield, as influenced by pH, temperature and extraction time, for the citric acid in skin absence

	Variables			Responses	
Run	**pH**	***T***	**ET**	y_{pec} (%)	
3	1.9 (0)	65 (0)	10 (−1)	48.85	48.99
7c*	1.9 (0)	65 (0)	45 (0)	43.12	42.74
4	1.9 (0)	65 (0)	90 (+1)	61.58	61.96
5	1.9 (0)	90 (+1)	45 (0)	39.28	39.64
2	1.9 (0)	40 (−1)	45 (0)	39.57	39.13
6	2.6 (+1)	65 (0)	45 (0)	29.32	29.68
1	1.2 (−1)	65 (0)	45 (0)	61.22	61.32

*Central point of the design.

The linear coefficients for pH and the linear and quadratic coefficients for *T* and ET influenced the pectin extraction significantly. For pH, the linear regression coefficients were negative, indicating that a higher acid concentration contributes positively to a higher pectin yield. The linear regression coefficients for *T* and ET were positive, indicating that a better pectin yield was obtained at higher temperatures and extraction times, respectively. However, as shown by the T^2 coefficient, for low temperatures, the responses decrease quadratically, while for the coefficient ET^2, for a long time, the responses increase quadratically. As obtained in full factorial design, the coefficient of the interaction of *T* and ET was again significant and negative. This interaction can be observed in Fig. 1 by plotting of marginal means analysis. This plot shows that within a high level of *T*, the response is significantly higher at the

Table 4 Coded coefficients of the central composite design model used to predict pectin yield extracted by citric acid

	Coeff.	SE	*t*	*P*
Mean	47.40*	1.89	25.0	0.000
pH	−13.71*	1.10	−12.0	0.000
pH^2	−2.68	2.17	−1.2	0.230
T	7.06*	1.10	6.4	3.00e-6
T^2	−8.66*	2.17	−4.0	7.00e-4
ET	3.80*	1.10	3.4	0.003
ET^2	6.91*	2.22	3.1	0.005
pH–*T*	0.75	1.23	0.6	0.551
pH–ET	−0.14	1.23	−0.1	0.909
T–ET	−3.28*	1.23	−2.7	0.015
R^2	0.945			

*Significant coefficients. df = 20, α = 0.05.

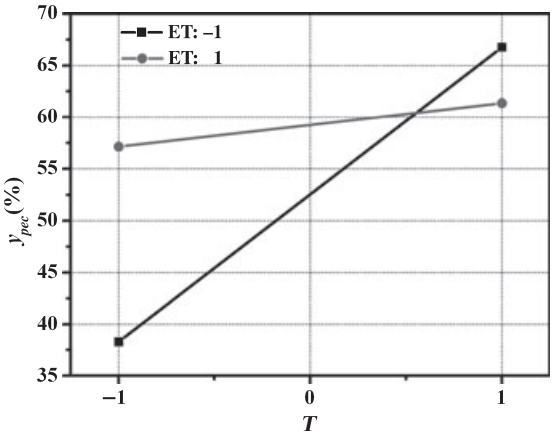

Figure 1 Plot of marginal means analysis for the interaction of temperature (*T*) and extraction time (ET), with ET varying within different *T* levels.

negative level of ET. This result suggests the use of shorter extraction times and higher temperatures.

A graphic representation of the models obtained for citric acid can be seen in Fig. 2. The measured vs. predicted values plot (Fig. 2a) for pectin yield shows a good fit using the quadratic model. The correlation coefficient R^2 was 0.945. The residuals plot vs. measured pectin yield values (Fig. 2b) shows that there is a random behaviour and there does not appear to be any regular trend. Thus, it can be assumed that the normality, independence and randomness of the residuals were satisfied (Teófilo & Ferreira, 2006).

The coded model for citric acid is in eqn 10, while Fig. 3 shows the response surface elaborated from the decoded regression model, keeping the extraction time fixed at 10 min (level -1). This surface shows that significant increase in the pectin yield is obtained when the variables pH and *T* are used at their levels -1 (pH 1.2) and +1 (80–90 °C), respectively. In this sense, it can be concluded that, for the citric acid, either with the presence or absence of skin, at a temperature of approximately 90 °C, with a pH of approximately 1.2 and an extraction time of approximately 10 min, the best pectin yield will be obtained.

$$y_{\mathrm{pec}}(\%) = 36.69 - 13.85\mathrm{pH} + 10.34T - 8.66T^2 + 0.75\mathrm{pH} \cdot T \quad (10)$$

The best conditions for maximisation of pectin yield (70%) were the use of citric acid at 80 °C, pH1 with an extraction time of 10 min considering the model extrapolation. The ET level obtained with citric acid was satisfactorily low resulting in energy saving with respect to the extractions. The pH levels indicate that the interaction between pectin and citric acid molecules is fundamentally important, as the pH values for other acids were slightly larger. These results are similar to those found by Pagán & Ibarz (1999) and Pagán *et al.* (2001) who, by extraction of pectin from fresh and stored peach pomace, respectively, verified that for a constant time, as temperature increased and pH decreased, the yield increased.

According to Calliari & Gómez (2004), the maximum yield obtained for pectin extraction from orange pulp with citric acid was 77%, with a time of 2 h at 100 °C. Virk & Sogi (2004) studied the extraction of pectin from apple peel, where the maximum yield (78%) was obtained using 1% citric acid.

Pectin characterisation

The DM value of extracted pectin with citric acid in the optimised condition (45.94%) was slightly higher than that of commercial LMP. This DM was close to those determined in the yellow passion fruit rind by Yapo & Koffi (2006), using water, ammonium oxalate and dilute acid solutions as extractor. Virk & Sogi (2004) also obtained LMP (33.44%), extracted from apple peel, with citric acid as extractor.

The GalA contents of CEP and LMP were 68.7% and 54.1%, respectively. FCC (Food Chemical Codex) and FAO (Food and Agriculture Organisation), European Union (Willats *et al.*, 2006) stipulate that 'pectin' must consist of at least 65% GalA. The amount of GalA of CEP indicates that the extraction of pectin from passion fruit peel using citric acid as extractor was effective.

The acetyl value obtained by titrimetry of CEP was 0.3%. This value is consistent with that obtained by the HPLC method for passion fruit rind pectin (0.3–0.5%), as reported by Yapo & Koffi (2006). Virk & Sogi (2004) obtained acetyl value of 0.7% for apple peel pectin extracted by citric acid. No acetyl groups were found in the commercial citrus LMP.

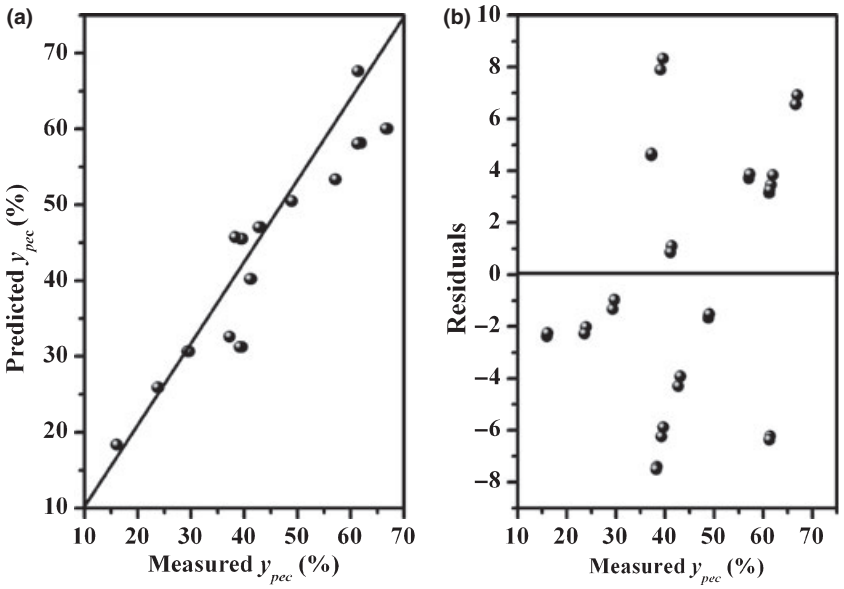

Figure 2 (a) Plot of the predicted vs. observed pectin yield; and (b) plot of residual vs. observed pectin yield for the citric acid.

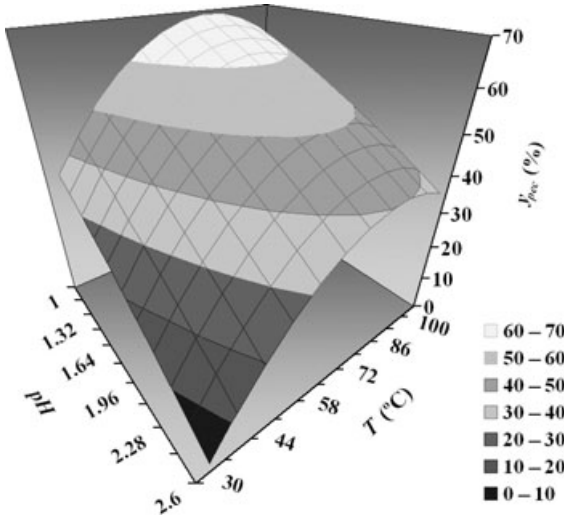

Figure 3 Response surface of the pectin yield using citric acid as extractor with extraction time as in eqn 9.

Conclusion

No statistical difference between the samples with and without skins was observed with citric acid using the screening experimental design. For the strong acids, hydrochloric and nitric acids, the pectin yields were 26% and 38%, respectively, and were obtained using increased time and higher temperature conditions. The best pectin yield (70%) was obtained for citric acid with extraction conditions optimised (pH 1.0, 80 °C and 10 min) using RSM. The extracted pectins with citric acid were rich in anhydrogalacturonic acid and had a low DM. These results demonstrate the successful extraction of pectin with citric acid, providing potential benefits for industrial extraction of pectin from an economic and environmental point of view.

Acknowledgments

The authors acknowledge CNPq for financial support and Dr Carol H. Collins for English revision.

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