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Optimization of extraction of high-ester pectin from passion fruit peel (*Passiflora edulis* flavicarpa) with citric acid by using response surface methodology

Eloísa Rovaris Pinheiro [a], Iolanda M.D.A. Silva [a], Luciano V. Gonzaga [a], Edna R. Amante [a], Reinaldo F. Teófilo [b], Márcia M.C. Ferreira [b], Renata D.M.C. Amboni [a,*]

[a] *Departamento de Ciências e Tecnologia de Alimentos, Centro de Ciências Agrárias, Universidade Federal de Santa Catarina, Rod. Admar Gonzaga, 1346, Itacorubi, 88034-001 Florianópolis, SC, Brazil*
[b] *Instituto de Química, Universidade Estadual de Campinas, UNICAMP, 13084-971, P.O. Box 6154, Campinas, SP, Brazil*

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Abstract

A central composite design was employed to optimize the extraction of pectin with citric acid. The independent variables were citric acid concentration (0.086–2.91% w/v) and extraction time (17–102 min). The combined effect of these variables on the degree of esterification was investigated. Results have shown that the generated regression models adequately explained the data variation and significantly represented the actual relationship between the independent variables and the responses. Besides that, the citric acid concentration was the most important factor to affect the degree of esterification, as it exerted a significant influence on the dependent variable. Lower citric acid concentration increased the pectin degree of esterification. The surface response showed the relationships between the independent variables, and thus responses were generated. Through this surface, the satisfactory condition of 0.086% w/v citric acid for 60 min was established for extraction of high-ester yellow passion fruit pectin.

Keywords: Pectin extraction; Passion fruit peel; Degree of esterification; Response surface methodology; Central composite design

1. Introduction

Pectin is a high-value functional food ingredient widely used as gelling agent and stabilizer. It is also an abundant, ubiquitous and multifunctional component of the cell walls of all land plants (Willats et al., 2006).

The main raw materials used to produce commercial pectin are apple pomace and citrus peels (May, 1990). Very little is known about the pectic substances in passion fruit in general. The two main edible species cultivated for commercial purposes are the purple passion fruit (*P. edulis* Sims), typically consumed fresh due to its sweeter taste, and the yellow passion fruit or 'maracuja' (*P. edulis* f. flavicarpa Degener), commonly used for production of juice; either pure or sweetened due to its slightly acidic taste (Yapo and Koffi, 2006). The peel represents about half of the fruit mass. Because of significant juice production, the peels, as a major waste, have become a substantial burden to the environment. Hence it is necessary to find a feasible way to turn the peels into useful products or to adequately dispose of them, seeking a positive environmental impact (Liu et al., 2006).

According to May (1999), only a few source materials have been used for commercial production of pectin as food additive. One of the reasons for this is that most of the pectic materials present in nature do not have any functional properties; in particular, the ability to form sugar acid gel systems, and this property has been the main requirement of commercial pectin until recently.

* Corresponding author. Tel.: +55 48 3721 5384; fax: +55 48 3721 9943.
E-mail address: ramboni@cca.ufsc.br (R.D.M.C. Amboni).

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Pectin extraction is a multiple-stage physicochemical process in which the 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 (Pagán et al., 2001). Reports are available on the extraction of pectins with mineral acids, such as sulfuric acid (Yapo et al., 2007; Yapo et al., 2007), hydrochloric acid (Iglesias and Lozano, 2004; Kratchanova et al., 2004; Mesbahi et al., 2005; Fishman et al., 2006; Faravash and Ashtiani, in press), nitric acid (Pagán et al., 2001; Yapo and Koffi, 2006), and tartaric acid (Canteri-Schemin et al., 2005). However, very little is known about the extraction of pectin with citric acid (Virk and Sogi, 2004; Canteri-Schemin et al., 2005; Marcon et al., 2005), that could be better than the other extractors from an economic as from an environmental point of view.

Pectin is a polymer of α-galacturonic acid with a variable number of methyl ester groups (Liu et al., 2006). However, pectin also contains α-L- rhamnopyranosyl residues in the backbone chain and branch chains of arabinan and galactan, and their fine structure vary considerably (Shingthong et al., 2004).

Some of the carboxylic groups of galacturonic acid molecules in the pectin chains are methyl esterified and the percentage of esterified groups is expressed as DE (degree of esterification). Depending on the degree of esterification, pectin is divided into two major groups: high-ester pectin, with DE higher than 50%, and low-ester pectin, with DE lower than 50% (Thakur et al., 1997). In high-ester pectin, the junction zones are formed by the cross-linking of homogalacturan through hydrogen bond and the hydrophobic interaction between methoxyl groups, both of which are promoted by high-sugar concentration and low pH. In low-ester pectin, junction zones are formed by calcium cross-linking between free carboxyl groups (Willats et al., 2006).

The main objective was to develop an approach that would bring a better understanding of the relationships between the variables (citric acid concentration and time of extraction) and the response (degree of esterification); and to obtain optimum conditions for pectin extraction from passion fruit peel. Thus a response surface methodology (RSM) using central composite design was employed. The advantage of this methodology is the simultaneous investigation of the main factors from a small number of experiments. The investigated region can lead to optimum pectin extraction conditions.

2. Methods

Passion fruits (*Passiflora edulis* flavicarpa) were obtained from the CEASA fruit farm, Florianópolis, Brazil, from March to May 2005. The fruits at the same ripening stage and with similar peel colours were selected. All the chemical reagents used were of analytical grade.

2.1. Preparation and chemical analysis of passion fruit peel flour

The passion fruit was washed and the pulp was separated from the flesh. The pulp was not studied at all. The skin (flavedo) was removed and the peels were dried in a air-circulate oven (Model 171, FABBE, São Paulo, Brazil) at 55 °C until their weight was constant. The dried peels were then milled to a dry 60 mesh size powdered passion fruit peel and the resulting product, referred to as 'passion fruit peel flour', was used as the raw material for all the pectin extraction and characterization assays. The passion fruit peel flour was packaged in a polyethylene bag and stored in a freezer (-18 ± 2 °C) until required.

The passion fruit peel flour was analysed for moisture, lipid, crude protein ($N \times 6.25$), total ash contents (AOAC, 1998), and soluble and insoluble dietary fibre content (AACC, 1999), expressed as g/100 g (dry basis). All the analyses were carried out in triplicate ($n = 3$). Total carbohydrate was calculated by difference.

2.2. Pectin extraction

The extraction procedure was according to the Canteri-Schemin et al. (2005) method, with slight modification. Pectin was extracted with different citric acid concentrations and extraction times, under reflux in a condensation system at 97 °C (solute/solvent 1:50). 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 (containing pectin) was centrifuged for 30 min at 6000 rpm. The supernatant was precipitate with absolute ethanol (1:2 v/v) and then left to rest for one hour in order to allow pectin flotation (Kalapathy and Proctor, 2001). The floating pectin was separated by filtration (# 40 filter paper) and rinsed with absolute ethanol. The pectin produced was dried in an air-circulate oven at 45 °C for 12 h. The resulting material was milled to a dry 60 mesh size powdered pectin.

2.3. Determination of degree of esterification

The degree of esterification (DE) of pectin samples were determined by the potentiometric titration method by Bochek et al. (2001). Dried pectin (0.2 g) was placed in a weighing bottle for titration and wetted with ethanol. Distilled water, at 40 °C (20 mL), was added by stirring. The polymer was dissolved by stirring for 2 h. The resulting solution was titrated with 0.1 N NaOH in the presence of phenolphthalein and the result was recorded as the initial titre (It). Then, a 0.1 N NaOH solution (10 mL) was added to a neutralized polygalacturonic acid sample after determination of the free carboxy groups. The weighing bottle was plugged with a stopper. The content was stirred at room temperature for 2 h to saponify the esterified carboxy