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Extraction of pectin from passion fruit peel (*Passiflora edulis* f. *flavicarpa*) by microwave-induced heating

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ABSTRACT

Pectin is a heteropolysaccharide obtained from primary cell walls of terrestrial plants, which is a very important raw material for food and pharmaceutical products. Extraction of pectin from the peels of yellow passion fruit (*Passiflora edulis* f. *flavicarpa*) under microwave-induced heating was investigated in the present study. Three types of acids (tartaric, acetic and nitric acid) were employed as extracting agents. The effect of extraction time and microwave-power on yield of pectin has been studied using the response surface methodology. The results indicate that exposure time and microwave-power significantly affects the yield of pectin extraction with both nitric and tartaric acids. However, the extractions using acetic acid were significantly affected only by the exposure time. For all scenarios, the highest yields were obtained when the highest levels of power and time were used (628 W and 9 min). Under these conditions, the yield of pectin obtained with nitric and acetic acids were 13 and 12.9% respectively. Tartaric acid emerged as the best extracting agent in terms of yield (18.2%), however, the obtained pectin exhibited low purity and low degree of esterification. Pectin extracted from passion fruit by employing acetic and nitric acid presented better properties: high molar mass (4.625×10^5 for acetic acid and 4.966×10^5 for nitric acid), degree of esterification (64.56% for acetic acid and 64.15% for nitric acid) and content of uronic acids (62.5% for acetic acid and 82.3% for nitric acid).

1. Introduction

Passion fruit (*Passiflora edulis* f. *flavicarpa*) being a good source of vitamin C is commonly employed in juice processing. The annual production of this juice in Brazil alone is estimated to be more than 920,158 tons in 2010 (IBGE, 2013). Such a wide-scale use of this fruit, inevitably leads to the generation of vast quantities of the fruit peels, which constitutes about half of the fruit mass. These fruit peels are discarded as a major waste, causing a substantial burden on the environment. It is therefore imperative to find adequate disposal of these peels or means to convert the peels into useful products (Liu, Shi, & Langrish, 2006; Pinheiro et al., 2008). Recent studies have shown that the pericarp (integral part of the peel) of passion fruit, even though processed and stored, could be used as raw material for obtaining co-products in food industry, such as dietary fiber and other bioactive compounds (Yapo, 2009a; Yapo & Koffi, 2006).

Pectin is a polysaccharide found ubiquitously in cell walls of all plants (Carpita & McCann, 2000). This polysaccharide is mainly composed of two moieties (i) homogalacturonan made up of (1-4) linked α-D-galacturonic acid and (ii) rhamnogalacturonan I consisting of (1-2) repeating linked, α-L-rhamnose-(1-4) and α-D-galacturonic acid disaccharide. Rhamnogalacturonan I usually contains neutral side chains of arabinan, galactan or arabinogalactan.

The ratio of esterified carboxylic acid units to total carboxylic acid units in pectin is termed the degree of esterification (DE), which has a major influence on gel properties of pectin. Depending on DE, pectin is commercially divided into two major groups: high-ester pectin, with DE higher than 50%, and low-ester pectin, with DE lower than 50% (Thakur, Singh, & Handa, 1997).

Pectin represents a high-value functional food ingredient widely used as gelling agent and stabilizer, particularly in jams and jellies (Willats, Knox, & Mikkelsen, 2006). Pectin extraction is a multiple-stage physicochemical process which involves hydrolysis and extraction of pectin macromolecules from plant tissue, purification of the liquid extract and isolation of the extracted pectin from the liquid. These processes are influenced by various factors, mainly temperature, pH, and time (Pagán, Ibarz, Llorca, Pagán, & Barbosa-

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Cánovas, 2001). Usually the traditional technique requires longer extraction time thus running the severe risk of thermal degradation for the thermolabile materials. Conventional techniques for the extraction of constituents are time and solvent consuming and thermally unsafe. Keeping in pace with such requirements recent times has witnessed the use and growth of new extraction techniques with shortened extraction time, reduced solvent consumption, increased pollution prevention concern and with special care for thermolabile constituents (Luque de Castro & Garcia-Ayuso, 1998; Mandal, Mohan, & Hemalatha, 2007). Although it has been shown that the pericarp of passion fruit can be used as raw material for pectin isolation (Kliemann et al., 2009; Pinheiro et al., 2008; Yapo, 2009a, 2009b; Yapo & Koffi, 2006), the extraction under microwave heating was not investigated.

The use of microwave for extraction of constituents from plant material has shown tremendous research interest and potential. There are two types of microwave systems currently used: closed extraction vessels and focused microwave ovens. The latter, used in this work, is also named as solvent extraction, in which only a part of the extraction vessel containing the sample is irradiated with microwave, this system operates in atmospheric pressure (open-vessel). The use of atmospheric pressure substantial provides advantages over pressurized vessels such as: open-vessel operation is more suitable with thermolabile species as it uses low temperatures relative to closed vessel systems; the low cost of the equipment required; the absence of any requirement for cooling down or depressurization; the ability to add reagents at any time during the treatment, among others (Mandal et al., 2007).

In addition, conventionally, pectin is extracted from citrus peels and apple pomace by employing strong mineral acids (May, 1990; Rolin, 1993). The use of such strong acids in the extraction step leads to corrosion of equipment and has deleterious effects on the environment. The need for replacement of these extracting agents with milder weak organic acids could be a potential means to minimize aforementioned adverse effects. As the yield and composition of pectins depends on the conditions used during pectin isolation and purification (Levigne, Ralet, & Thibault, 2002; Rolin, 1993), in this work pectins from yellow passion fruit peels were extracted using different acids under microwave heating.

The main aim of this study was to employ two weak organic acids, such as tartaric and acetic, and the nitric acid for extraction of pectin from peels of yellow passion fruit under microwave heating. The factors influencing the yield of pectin such as intensity of electromagnetic field and exposure time were also examined. The extracted pectin should be of high DE, purity and molecular weight and the yield should be as high as possible.

2. Materials and methods

Samples of yellow passion fruit peel (*P. edulis* f. *flavicarpa*) were obtained from the pulps producing industry located in Paraná, Brazil. These fruit peel represents a waste generated during the fruit processing operations. These peels were composed of an intact pericarp. The commercial citrus pectin, employed for comparative analysis was procured from Vetec P.A., Brazil. All chemical reagents employed in this study were of analytical grade.

2.1. Preparation of passion fruit peel flour (PFF)

The fruit peels were thoroughly washed in running water and subjected to a blanching treatment, which results in enzymatic inactivation. For this, the peels were immersed in water at a temperature of 97 °C for 3 min, then, the peels were transferred to another water bath at room temperature and maintained for 15 min. Subsequently, the peels were dried in an air-circulated oven at 50 °C until a constant weight was obtained. The dried peels were then milled to 60 mesh size powdered passion fruit peel and the resulting product, referred to as "passion fruit peel flour (PFF)". The passion fruit peel flour was used as raw material for pectin extraction. The passion fruit peel flour was packaged in a polyethylene bag and stored in a freezer (−15 ± 2 °C) until required.

2.2. Pectin extraction

Pectin was extracted using a modified version of the method presented elsewhere by Canteri-Schemin, Fertonani, Waszczynskyj, and Wosiacki (2005). The extraction process was carried out in 600 mL beakers, followed by heating in a microwave oven (CCE model M − 304). 4 g of passion fruit peel flour was added to 100 mL of distilled water in a beaker. This was followed by addition of 100 mL of an acidic solution, with concentration adjusted so as to maintain a final pH of 2 for the solutions. The beaker was partially covered with a glass lid and then subjected to microwave heating for predetermined time intervals (3, 6 and 9 min). As the extraction system remained partially open, gradual evaporation of the solvent was observed. In order to maintain a constant suspension concentration, the amount of evaporated water was replenished to the system at regular intervals.

The suspension, still warm, was vacuum filtered in synthetic fabrics; the retentate was discarded and the filtrate (containing the soluble pectin) was cooled to 4 °C. To isolate the soluble pectins from the filtrate, the extracted liquid was slowly added under magnetic stirring to two volumes of absolute ethyl alcohol, both maintained at 4 °C. This mixture was stirred for 10 min, after which it was allowed to rest for 30 min to facilitate the flotation of the pectin. The pectin thus obtained, was separated by vacuum filtration on a filter paper. The extracted pectin in a gel form was immersed in absolute ethyl alcohol for about 12 h and then was partially dehydrated by immersion in acetone for a few minutes. This was followed by drying pectin in an air-circulated oven at 40 °C until constant weight was obtained (approximately five hours). The resulting material was milled to dry powdered pectin. The extraction process of pectin is schematized in Fig. 1.

2.3. Experimental design

Response surface methodology was used to determine the optimum condition for pectin extraction from passion fruit peel flour. In order to evaluate the effect that the variables *extraction time* (tEx) and *microwave power* (*P*) present on the *yield of pectin extracted* (response) a complete factorial experimental design 2^2 was elaborated with the insertion of a central point. Experiments in the centre of the design were performed in order to make the estimation of pure error possible. The variable extraction time was evaluated at the levels 3, 6 and 9 min and the microwave power was evaluated at levels 356, 450 and 627.9 W. The pH of the solution was maintained constant (2.0) for the three kinds of acids studied. The complete design consisted of 4 experiments for each type of acid examined and three replicates in center point (Table 1). All the tests were conducted in duplicate at random. The *t*-Student test was performed with a significance level of 5%. For data analysis was used the software Statistica 10.

2.4. Degree of esterification

The degree of esterification (DE) of pectin samples were determined by potentiometric titration, as described by Bochek, Zabivalova, and Petropavlovskii (2001). The percentage of gravimetric yield of pectin was calculated from the ratio of the mass of dried pectin extracted and the mass of flour used as raw material.