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# Extraction of pectin from passion fruit peel assisted by ultrasound

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ABSTRACT

The effect of power intensity of ultrasound and temperature on the pectin extraction from passion fruit peel was evaluated by response surface methodology. The extraction was performed using a dried peel/extractant ratio fixed at 1:30 for 10 min of sonication. The dependent variables were the yield, the galacturonic acid content and the esterification degree of the extracted pectin. The highest yield of pectin was obtained by power intensity of 644 W/cm$^2$ and temperature of 85 °C. Under these conditions, the yield, the galacturonic acid content and the esterification degree were 12.67%, 66.65% and 60.36%, respectively. The conventional extraction (dried peel/extractant ratio 1:30, 10 min and 85 °C) was performed to compare the results to the ultrasound-assisted extraction. The results showed that the use of ultrasound promoted a better extraction yield of pectin when compared to the conventional method.



## 1. Introduction

Many compounds can be extracted from vegetable tissues and the efficiency of this process is measured both by the extraction yield and the minimal impact on the properties of the extracted compound. The conventional heating extraction is time consuming and uses large amount of solvents (Wang & Weller, 2006; Tiwari, 2015). A number of up-to-date alternatives to conventional techniques have been proposed for the extraction of target compounds from various vegetables sources, including ultrasound (Barrales, Rezende, & Martínez, 2015), moderate electric field (de Oliveira, Giordani, Gurak, Cladera-Olivera, & Marczak, 2015), pulsed electric field (Medina-Meza & Barbosa-Cánovas, 2015) and high pressure These up-to-date techniques offer potential to improve process efficiency, to enhance the extraction yield and to improve the quality of the extracted compound. The low temperature used in these techniques is also an important characteristic, since it can decrease the thermal degradation of extracted compounds. This work will focus on the ultrasound technique, which is an innovative, clean and green extraction technology for various molecules and biomaterials, including bioactive molecules, proteins, peptides, essential oils and polysaccharides.

Pectin is one of the most important and widely used polysaccharide in food industry. Pectin consists of a backbone of α-(1,4) galacturonic acid residues which are partially esterified with methyl alcohol or acetic acid in carboxylic acid and it can be extracted from peels of fruits. This soluble fiber is used as a gelling, stabilizing and thickening agent in food systems such as jams and jellies, confectionery and fruit juice (Thakur, Singh, & Handa, 1997). Besides, pectin is suggested to have various pharmaceutical activities, including wound healing, lipase inhibition, apoptosis induction of human cancer cell, as well as immunostimulating, antimetastasis, anti-ulcer and cholesterol decreasing effects (Espinal-Ruiz, Restrepo-Sánchez, Narváez-Cuenca, & McClements, 2015).

Passion fruit (*Passiflora edulis Sims f. flavicarpa Degener*) peel (mesocarp and epicarp) represents 60% of fruit (in mass) and contains a large amount of bioactive compounds and polysaccharides, such as pectin (López-Vargas, Fernández-López, Pérez-Álvarez, & Viuda-Martos, 2013). According to some authors, the content of pectin in passion fruit peel ranges from 15 to 20 g/100 g dry mass (Kliemann et al., 2009 Pinheiro et al., 2008; Seixas et al., 2014). The traditional method for pectin extraction includes

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direct boiling (60 °C −100 °C) for 20–360 min and low pH (1.0–3.0) (Liu, Shi, & Langrish, 2006; Levigne, Ralet, & Thibault, 2002; Koubala et al. 2008). This process is time consuming, it can cause thermal degradation of the fiber extracted and the pectin yield is sometimes limited by the process conditions (Yeoh, Shi, & Langrish, 2008).

Ultrasound-assisted extraction is a process that uses acoustic energy and solvents to extract target compounds from various plant matrices. The enhancement of the mass transfer is brought about by the acoustic cavitation induced in a liquid medium, which is one of the beneficial effects of this technology (W. Wang et al., 2015). As pectin is a soluble fiber, which is presented in the plant cell walls, cavitation and cell disruption caused by ultrasound waves may enhance the mass transfer from the solid matrix to the solvent improving the pectin extraction.

The aim of this study is to evaluate the potential of the ultrasound-assisted extraction of pectin from passion fruit peel, using nitric acid as the extracting agent. For this purpose, an experimental design was applied considering the extraction temperature and the power intensity as independent variables.

## 2. Materials and methods

### 2.1. Plant material – passion fruit powder

Two batches of yellow passion fruit (*P. edulis Sims* f. *flavicarpa Degener*) were purchased from a local market (Porto Alegre, RS, Brazil). The fruits were washed and sliced to remove the pulp and the seeds. The peels (composed of mesocarp and epicarp) were immersed in water at 100 °C for 3 min followed by cooling in an ice bath (blanching). The peels were grounded in a domestic blender and the peel was dried in an oven with air circulation at 60 °C for 72 h. The dried peels were then milled and passed through of a sieve of 0.250 mm. The powders of passion fruit peel obtained from the two different batches were thoroughly mixed and all further experiments were performed using samples from this mixture. The final moisture content of the powder was 6.34 ± 0.76 g 100 $g^{-1}$.

### 2.2. Pectin extraction

The extraction process was divided in three different steps, as described bellow.

#### 2.2.1. Determination of sonication time for pectin extraction assisted by ultrasound

The extraction process was performed using the following conditions, according to previous studies described by de Oliveira et al. (2015): dried peel/solvent ratio 1:30 (g/mL) and pH 2.0 (1.0 M $HNO_3$). The power intensity of the ultrasound was 664 W/$cm^2$ (maximum power of the equipment) and the frequency was 20 kHz. The temperature was maintained below 50 °C since the purpose of this step was to observe the effect of the ultrasound during different times of extraction (previous experiments showed that the pectin started to be extracted with temperatures higher than 50 °C). The temperature was controlled by a thermostatic bath (Lauda R. Wobser GmbH & Co. KG, modelo Alpha RA 12, Lauda-Königshofen,Germany). Five different moments were evaluated: 3, 6, 10, 15 and 20 min, chosen after preliminary tests. The passion fruit peel powder (6.6 g) was solubilized in 200 mL of acid solution ($HNO_3$ - pH 2.0) and put in the extraction cell, a vessel, made of Pyrex glass, with a water jacket and a volume of 300 mL, with the ultrasound probe (1.2 cm of diameter and 750 W) (Sonics & Materials, Inc., model VCX750, Newtown, USA). The processed sample was centrifuged and the supernatant was filtered using vacuum filtration. The filtrate was collected and stored in a refrigerator at 4 °C for 30 min for subsequent purification (described below). All experiments were performed in triplicate.

#### 2.2.2. Determination of power intensity and temperature for extraction of pectin assisted by ultrasound

This part of the study used a response surface methodology. In order to evaluate the effect of the variables power intensity and temperature on the yield of the extraction, galacturonic acid content and esterification degree of pectin, a factorial experimental design $2^2$ with three replications of the center points was performed. The temperature ranged from 45 to 85 °C and power intensity ranged from 132.8 to 664.0 w/$cm^2$. The pH of the solution (pH 2.0; 1.0 mol/L $HNO_3$), peel/solvent ratio 1:30 (g/mL), time (10 min that were determined in the first step) and frequency (20 kHz) were maintained constant. In developing the regression equation, the test factors were coded according to the following equation:

$$x_i = \frac{(X_i - X_0)}{\Delta X_i} \quad (1)$$

where $x_i$ is the coded value of the independent variable, $X_i$ the natural value of the independent variable, $X_0$ the natural value of the independent variable at the center point and $\Delta X$ the step change value (the value of $\Delta X$ was 20 °C for temperature and 256.6 W/$cm^2$ for power intensity). The model regression equation for the predicted response $Y$ is:

$$Y = b_0 + b_1x_1 + b_2x_2 + b_{12}x_1x_2 \quad (2)$$

The results were analyzed by the Experimental Design Module of the Statistica 8.0 software (Statsoft, USA).

#### 2.2.3. Extraction of pectin by conventional heating

Conventional extraction (performed in three replicates) was conducted in order to compare to the results obtained by ultrasound extraction. Pectin extracted by the conventional heating was based on the method of (Kratchanova, Pavlova, & Panchev, 2004) with slight modifications. The following conditions were maintained constant: pH at 2.0 (1.0 mol/L HNO3), peel/solvent ratio 1:30, temperature of 85 ± 2 °C and 10 min (same time used in an ultrasound extraction). After the time of extraction, the hot mixture was centrifuged (1500 × g) and filtered by vacuum filtration, and the filtrate was collected and stored in a refrigerator at 4 °C for subsequent purification.

#### 2.2.4. Pectin purification procedure

Extracted pectin was precipitated using a solution of ethanol (190 mL) and distilled water (10 mL) at 4 °C for 30 min. The precipitated pectin was separated by vacuum filtration and then immersed in a solution of ethanol (70 mL) and distilled water (30 mL) for 12 h to remove the impurities (such as monosaccharides, disaccharides and phenolic compounds) according to Minkov, Minchev, & Paev, 1996. The pectin was washed with acetone and immediately dried at 40 °C for 12 h in an oven with air circulation (until constant weight). Values are expressed in dry matter. The extraction yields ($Y$) were calculated according to the following equation:

$$Y = \frac{Pectin\ Weight(d.m)}{Sample\ Weight(d.m)} \times 100 \quad (3)$$