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Optimization of microwave assisted extraction of pectin from orange peel

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ABSTRACT

In this study, microwave-assisted extraction was applied for pectin extraction from the dried orange peel and Box–Behnken response surface design was used to study and optimize the effects of processing variables (microwave power, irradiation time, pH and solid–liquid ratio) on the yield of pectin. The amount of pectin extracted increased with increasing microwave power, while it reduces as the time, pH and solid–liquid ratio increased. From the results, second order polynomial model was developed and it adequately explained the data variation and significantly represented the actual relationship between independent variables and the response. An optimization study using Derringer's desired function methodology was performed and optimal conditions based on both individual and combinations of all independent variables (microwave power of 422 W, irradiation time of 169 s, pH of 1.4 and solid–liquid ratio of 1:16.9 g/ml) were determined with maximum pectin yield of 19.24%, which was confirmed through validation experiments.

1. Introduction

Citrus fruits are an important crop throughout the world and oranges account for approximately 65% of all the citrus fruits produced (Luengo, Álvarez, & Raso, 2013). Citrus fruits are mainly used as either fresh juice or packed drinks. Peel makes up roughly half of an orange's mass and is the main by-product from the industrial processing of this fruit. Wastes generated from citrus (Cerezal, Larrauri, & Pinera, 1995; Cho, Lee, & Kim, 2003; Contreras-Esquivel, Hours, Voget, & Mignone, 1999) processing industries are reported to be rich source of pectin. Pectin is a complex carbohydrate, which is a significant component of the primary cell wall of plants. Pectin is a polymer of α-galacturonic acid with a variable number of methyl ester groups. Pectin is a natural food additive used extensively in the food industry as a thickener, a texturiser, an emulsifier, stabilizer, gelling agent and other applications include fat replacers in spreads, salad dressings, ice cream and emulsified meat products (Liu, Shi, & Langrish, 2006). The world market demand for pectin is in excess of 30,000 tons annually and is growing by about 4–5% per annum (Yeoh, Shi, & Langrish, 2008). So, citrus-processing industries are commercially interested in recovering residual amounts of soluble solids after juice extraction. Therefore, it is necessary to establish a new method, by which the pectin could be extracted in a shorter time and in better quality.

Microwave assisted extraction (MAE) is an interesting alternative to conventional extraction methods and it has many advantages, such as shorter time, less solvent, higher extraction rate, better products with lower cost. Soxhlet method usually needs a few hours, even more than 20 h, while microwave-assisted extraction only needs a few minutes or seconds. Compared with supercritical fluid extraction, the apparatus of microwave-assisted extraction is simpler and cheaper (Hao, Han, Huang, Xue, & Deng, 2002). MAE increased the capillary-porous characteristics and the water absorption capacity of the plant material. These changes provide an opportunity for improving the extraction yield of target analytes from plant material (Kratchanova, Panchev, & Pavlova, 2004).

Pectin from orange peels has wide applications in the food industries. Since, the conversion of orange fruit peel into a valuable by product such as pectin offers great scope for utilization and also reduces the environmental pollution. Hence, the main objective of this study is to develop an MAE of pectin from orange peel and investigate the effect of process variables (microwave power, irradiation time, pH and solid–liquid ratio) and the response (pectin yield); and to obtain optimum conditions for maximum extraction yield of pectin from orange fruit peel. Thus, response surface

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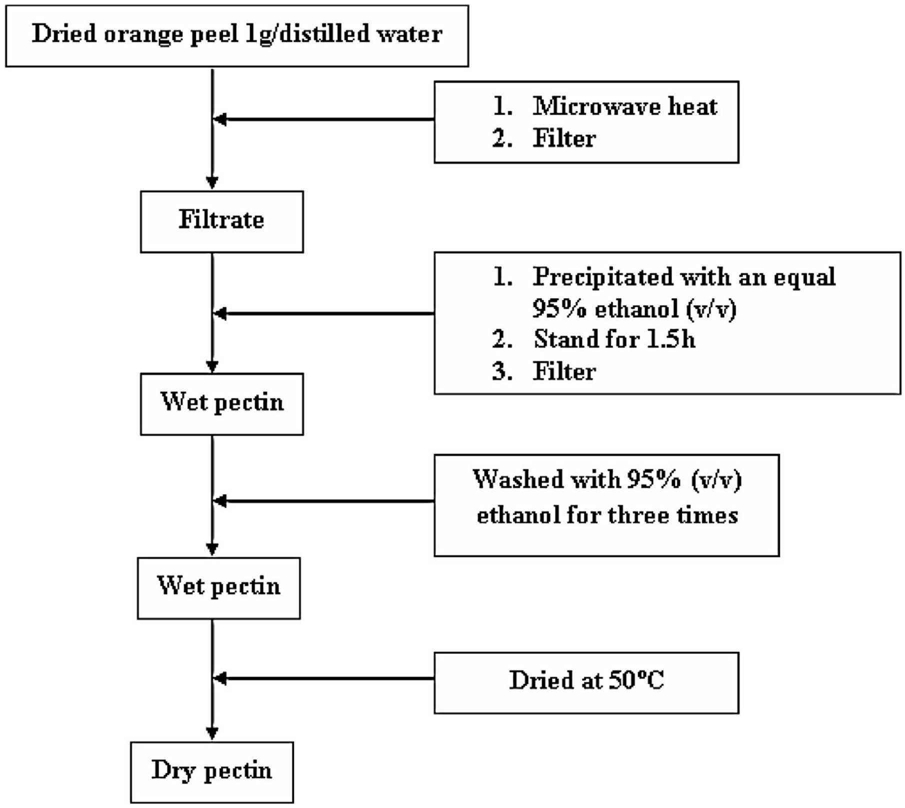

Fig. 1. Flow chart for pectin extraction from orange peel.

methodology (RSM) using Box–Behnken design was employed in this study. The advantage of this methodology is the simultaneous investigation of individual and interactive effects of process factors on the response from a small number of experiments (Prakash Maran, Sivakumar, Sridhar, & Thirgananasambandham, 2013).

2. Materials and methods

2.1. Raw materials and reagents

Orange (*Citrus reticulata*) fruit was obtained from local fruit orchard near Erode, Tamilnadu, India. The peels were removed, finely cutted and dried in a hot air oven (NSW, India) at 60 °C until it attains constant weight. Then the peels were pulverized and passed through a 40-mesh sieve to obtain powdered sample. The powder (moisture content of 12–14% on dry basis) was stored in bags and kept it dry environment prior to the experiments. All solvents and chemicals used in this study were analytical grade and purchased from Merk chemicals, Chennai, India.

2.2. Extraction of pectin from dried orange peel using MAE

MAE of pectin was performed according to the methods described by Wang et al. (2007) in an ordinary household microwave oven (LG, MH-3948 WB) at working frequency of 2450 MHz with adjustable microwave power and time. A flow chart for the extraction of pectin is depicted in Fig. 1. About 1 g of dried orange peel powder was weighed and placed into a 250 ml Pyrex beaker and different amount distilled water (10–20–30 ml) containing different pH values (1–1.5–2) was added. The pH of the solvent was adjusted with the help of sulfuric acid (0.05–0.016–0.005 mol/l). According to the experimental design (Table 1), the extraction was performed under different MAE conditions. The beaker was placed in the middle of the oven over a rotating dish and was exposed to microwave radiation at different powers (160–320–480 W) for the selected irradiation time (60–120–180 s). After microwave heating, the mixture in the Pyrex beaker was allowed to cool down to room temperature 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. The coagulated pectin mass was washed with 95% (v/v) ethanol for three times to remove the mono and disaccharides (Minkov, Minchev, & Paev, 1996).

2.3. Determination of pectin yield

After extraction, the wet pectin was dried at 50 °C in the hot air oven until its weight was constant and weighed. The pectin yield (PY) was calculated from the following equation proposed by Li, Jia, Wei, and Liu (2012).

$$PY = \left(\frac{m_0}{m}\right) \times 100 \quad (1)$$

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

2.4. Experimental design and statistical analysis

Experimental datas were analyzed using Design-Expert 8.0.7.1 (State-Ease Inc., Minneapolis, MN, USA) statistical package including ANOVA to obtain the interaction between the process variables and the response. A single factor experimental method was used to study the effect of process variables (microwave power, extraction time, pH and solid–liquid ratio) on the pectin yield from orange peel. According to the results from single factor experiments, a three level four factors Box–Behnken response surface