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Ultrasound-assisted extraction of pectin from sisal waste

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ABSTRACT

In this study, an efficient ultrasound-assisted extraction (UAE) of pectin from sisal waste was investigated and optimized. Response surface methodology (RSM) based on a three-level four-factor Box–Behnken response surface design (BBD) was employed to optimize the extraction conditions (ultrasonic power, extraction temperature, extraction time and solid–liquid ratio). Analysis of variance showed that the contribution of a quadratic model was significant for the pectin extraction yield. The experimental yield (29.32%) was obtained under the optimal condition (ultrasonic power of 61 W, temperature of 50 °C, time of 26 min and SL ratio of 1:28 g/ml) was well agreement with predicted values. Therefore, ultrasound-assisted extraction could be used as an alternative method to extract pectin from sisal waste with the advantages of lower extraction temperatures, shorter extraction time and reduced energy consumption.

1. Introduction

Sisal (*Agave sisalana*) is a monocotyledonous plant of great economic interest and grows well all round the year in hot climate and arid regions. Sisal products are widely used in marine industries and agriculture because of their strength, durability, stretchability, affinity for certain dyes, and resilience in saltwater. Some potential innovations include the use of the material as an organic fertilizer, a supplement in ruminant feed and a raw material in the production of medicine (Debnath, Pandey, Sharma, Thakur, & Lal, 2010). However, only 4% of the sisal leaves are used to create fibre. By-products from sisal extraction can be used for making biogas (Mshandete, Björnsson, Kivaisi, Rubindamayugi, & Mattiason, 2006), pharmaceutical ingredients (Santos & Branco, 2014) and polymer composites (Wu, 2011). The biomass left after fibres have been removed is now flushed away as waste. This sisal waste of material is composed of water (85%), parenchymal tissue, short fibres, primary and secondary metabolites and inorganic compounds (Silva & Beltrão, 1999). Currently, this waste is mainly land filled and dumped in nearby rivers, where microorganisms degrade it. As a result, the untreated waste causes consumption of oxygen in the recipient watercourses which leads to oxygen-deficient water zones with a negative effect on fish and other living organisms. Thus, recycling of sisal waste is crucial because it reduces environmental pollution and may also provide other highly useful and commercializable materials (Botura et al., 2013). This has led to focus on developing technologies aimed at determining applications for the remaining plant materials, especially the residue.

Recently, industrially, pectin is extracted from apple pomace, sugar beet pulp, and citrus peels using water acidified with a strong mineral acid, notably, nitric, hydrochloric or sulphuric acid (the so-called conventional acid extraction) with elevated temperature (Koubala et al., 2008). Acidic wastewater and environmental concerns make alternative extraction method to extract pectin from plant materials. In connection with the emerging concept of 'Green Chemistry', various novel methods including ultrasound-assisted extraction (UAE), supercritical fluid extraction (SFE), microwave assisted extraction (MAE) and accelerated solvent extraction (ASE) have been developed, as more environmentally friendly and energy efficient technologies, to enhance the recovery of valuable compounds from plant tissues (González-Centeno et al., 2014). Among them, UAE technology has received considerable attention due to its beneficial properties, involving shorten the extraction time, reduce the organic solvent waste, increase the extraction yield and enhance the quality of extracts when compared to conventional extraction (Tao, Wu, Zhang, & Sun, 2014). In fact, ultrasound has been recognized as an alternative approach to traditional extraction methods (Ebringerová & Hromádková, 2010; Rastogi, 2011; Awad, Moharram, Shaltout, Asker, & Youssef, 2012; Galanakis, 2013).

Some studies have indicated the presence of pectin in waste sisal leaves (Aspinall & Cañas-Rodriguez, 1958; Silva & Beltrão, 1999; Santos, Espeleta, Branco, & de Assis, 2013). However, the feasibility

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of using ultrasound for the extraction of pectin from sisal waste has not yet been explored in the literature. Hence, the objective of this present work is to investigate and optimize the effect of ultrasonic power, temperature, time and solid–liquid ratio on the extraction of pectin from sisal waste using four factors three level Box–Behnken response surface design. Box–Behnken design has proved to be an extremely valuable tool, it not only helps in determining the accurate optimum values of experimental parameters but also provides the possibility to evaluate the interaction between variables with a reduced number of experiments (Maran & Manikandan, 2012; Maran, Sivakumar, Thirgananasambandham, & Sridhar, 2014a). The optimized controlled conditions determined in this study should offer important reference values for any subsequent studies.

2. Materials and methods

2.1. Materials

Sisal waste utilized in this study was obtained from a sisal fibre processing unit near Chennai, Tamil Nadu, India. The sisal waste was dried in a hot air oven at 50 °C until it attains constant weight. The dried residuals were crushed into powder using a mill, stored in an air tight container and kept in dry environment prior to experiments. Analytical grade ethanol (99% purity) was purchased from Merck Chemicals, Mumbai.

2.2. UAE of pectin

Experiments were performed using an ultrasonic device (VCX 400, Sonics and Materials, USA and 0–400 W) with a 2.00 cm flat tip probe with provisions to set required output power, temperature and time. The extraction was performed by continuous ultrasound waves at a frequency of 20 kHz and ultrasonic generator probe was directly submerged into the suspension (10 g of dried power with appropriate volume of distilled water). The experiments were carried out in triplicates according to Table 1 and the average results were reported. During the extraction period, an amplitude controller was used to set the desired level of ultrasonic power and temperature was controlled at a desired level within ±1 °C. After extraction, the suspension was filtered using filter paper (Whatman no-1), centrifuged (5500 rpm for 15 min) 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 in order to remove the mono and disaccharides (Maran, Sivakumar, Thirgananasambandham, & Sridhar, 2013). After extraction, the wet pectin was subjected to drying at 50 °C in the hot air oven until it attains a constant weight. The pectin yield (PY) was calculated (dry basis) from the following method described by Maran, Sivakumar, Thirgananasambandham, and Sridhar (2014b).

$$\mathrm{PY}\,(\%) = \left(\frac{m_0}{m}\right) \times 100 \qquad (1)$$

where m_0 is the weight of dried pectin (g) and m is the weight of dried sisal waste powder (g).

2.3. Experimental design

In this study, four factors with three levels Box–Behnken response surface experimental design (BBD) was chosen to study and optimize the influence of process variables such as ultrasonic power (50–70 W), extraction temperature (40–60 °C), extraction time (10–30 min) and SL ratio (20–40 g/ml), on the maximum extraction yield of pectin from sisal waste. A total number of 29 experiments with five replicates (used to estimate experimental error) at the centre point were established and total number of experiments (N) was calculated from the following equation (Maran, Sivakumar, Sridhar, & Thirgananasambandham, 2013).

$$N = 2K(K-1) + C_0 \qquad (2)$$

where K is the number of factors and C_0 is the number of central point.

Experimental data were fitted to a second-order polynomial mathematical equation in order to express the relationship between independent variables and responses. The generalized form of second-order polynomial equation was given as follows:

$$Y = \beta_0 + \sum_{j=1}^{k}\beta_j X_j + \sum_{j=1}^{k}\beta_{jj} X_j^2 + \sum_{i}\sum_{i<j=2}^{k}\beta_{ij} X_i X_j \qquad (3)$$

where Y is the response; X_i and X_j are variables (i and j range from 1 to k); β_0 is the model intercept coefficient; β_j, β_{jj} and β_{ij} are interaction coefficients of linear, quadratic and the second-order terms, respectively; k is the number of independent parameters ($k = 4$ in this study) (Maran, Manikandan, Vigna Nivetha, & Dinesh, 2013). Statistical analysis of the experimental data was performed using the Stat Ease Design Expert 8.0.7.1 statistical software (Stat-Ease Inc., Minneapolis, USA).

2.4. Optimization and validation of optimized condition

Numerical optimization technique was adapted in this study to optimize the process conditions. For optimization of process variables, the regression model developed in this study was used to determine the optimal condition which could provide maximum pectin yield. The nature of the optimal condition (point of maximum or minimum or a saddle point response) was also evaluated by transforming the developed regression model into conical form and the Eigen values were computed using MATLAB software.

To determine the validity of optimized condition, additional triplicate experiments were performed under optimal conditions and average values of the experiments were compared with the predicted values of the optimized conditions in order to find out the accuracy and suitability of the optimized conditions.

3. Results and discussion

3.1. Experimental data analysis

The experimental data was fitted to various models (linear, interactive (2FI), quadratic and cubic) and the results were shown in Table 2. The results demonstrated that, linear and interactive (2FI) models exhibited lower R^2, adjusted R^2, predicted R^2 and also high p values, when compared with quadratic model. Cubic model was found to be aliased. Therefore the quadratic model incorporating linear, interactive and quadratic terms was chosen to describe and study the effect of process variables on the pectin yield from sisal waste.

3.2. Model fitting and statistical analysis

By applying multiple regression analysis on the experimental data, Design-Expert software generated the second-order polynomial equation which can express the relationship between process variables and the responses. The final equation obtained in terms of coded factors is given below.

$$\begin{aligned}\text{Pectin yield}\,(\%) &= 30.55 + 1.93X_1 - 2.15X_2 + 2.69X_3 - 0.24X_4 \\ &\quad - 0.59X_1X_2 - 0.052X_1X_3 - 1.52X_1X_4 + 2.92X_2X_3 + 1.36X_2X_4 \\ &\quad + 0.41X_3X_4 - 2.58X_1^2 - 5.81X_2^2 - 3.42X_3^2 - 2.43X_4^2 \end{aligned} \qquad (4)$$

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