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Optimization studies on acid hydrolysis of oil palm empty fruit bunch fiber for production of xylose

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Abstract

Oil palm empty fruit bunch fiber is a lignocellulosic waste from palm oil mills. It is a potential source of xylose which can be used as a raw material for production of xylitol, a high value product. The increasing interest on use of lignocellulosic waste for bioconversion to fuels and chemicals is justifiable as these materials are low cost, renewable and widespread sources of sugars. The objective of the present study was to determine the effect of H_2SO_4 concentration, reaction temperature and reaction time for production of xylose. Batch reactions were carried out under various reaction temperature, reaction time and acid concentrations and Response Surface Methodology (RSM) was followed to optimize the hydrolysis process in order to obtain high xylose yield. The optimum reaction temperature, reaction time and acid concentration found were 119 °C, 60 min and 2%, respectively. Under these conditions xylose yield and selectivity were found to be 91.27% and 17.97 g/g, respectively.

Keywords: Lignocellulose; Xylose; Oil palm empty fruit bunch (OPEFB); Acid hydrolysis; Optimization

1. Introduction

Bioconversion of lignocellulosic waste materials to chemicals and fuels are receiving interest as they are low cost, renewable and widespread in nature (Kuhad and Singh, 1993). Malaysia is well known for its potential in renewable resources such as oil palm waste, sugar cane bagasse and rice straw. In the process of extraction of palm oil from oil palm fruit, a lignocellulosic material oil palm empty fruit bunch (OPEFB) is generated as a waste product. Approximately fifteen million tons of OPEFB biomass waste is generated annually throughout Malaysia by palm oil mills. In practice this biomass is burned in incinerators by palm oil mills which creates environmental pollution problems in nearby localities. The OPEFB biomass contains cellulose, hemicellulose and lignin. It is estimated that OPEFB biomass is comprised of 24% xylan, a sugar polymer made of pentose sugar xylose. This xylose can be used as substrate for production of a wide variety of compounds by chemical and biochemical processes (Wyman, 1994; Almeida e Silva et al., 1995). One such compound is xylitol, which is extensively used in food, pharmaceutical and thin coating applications (Parajo et al., 1995; Torget et al., 1991). The most important application of xylitol is its use as an alternative sweetener in foods for diabetic patients (Pepper and Olinger, 1988). Other important uses of xylitol are: as an anticariogenic agent in tooth paste formulations, as thin coatings on chewing vitamin tablets, in mouth washes, in beverages and in bakery products (Makinen, 1976; Emodi, 1978; Hyvonen and Koivstoinen, 1982). Research investigations on dilute acid hydrolysis of various raw materials such as sugar cane bagasse, sorghum straw, corn cobs and eucalyptus wood have been carried out by several workers (Roberto et al., 1995; Herrera et al., 2003). From the research studies it was revealed that under controlled treatment conditions, acid hydrolysis of ligno-

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cellulosic biomass mainly produced xylose from xylan with the cellulosic and lignin fractions remaining unaltered. Xylan is more susceptible to hydrolysis by mild acid treatment due its amorphous structure compared to cellulose which needs severe treatment conditions for its crystalline nature. Although xylose was the main sugar obtained from hemicellulose, other byproducts such as glucose, acetic acid, furfural etc., were also produced in low amounts during the hydrolysis process (Domínguez et al., 1997; Silva et al., 1998). It was also reported that amount of sugar released during hydrolysis, depended on type of raw material and operating conditions of the experiment. Acid concentration is an important parameter for release of sugars whereas temperature is mainly responsible for degradation of sugars to various byproducts (Neureiter et al., 2002). Acetic acid and furfural are potential inhibitors to yeast metabolism. When these compounds are present in the hydrolysate, they inhibit the fermentation process by causing cell morphological change or ultimate death of the organism (Van Zyl et al., 1991). To keep the concentration of byproducts (glucose, acetic acid and furfural) in the hydrolysate at a low level it is necessary to run the hydrolysis reaction at less severe conditions.

To our knowledge, there are no reports on acid hydrolysis of OPEFB biomass. The aim of the present investigation was to determine the effect of acid concentration, reaction temperature and reaction time period on release of sugars (xylose and glucose) and formation of byproducts (acetic acid and furfural) from OPEFB biomass. To maximize the formation of xylose in hydrolysate optimization process was followed using response surface methodology (RSM) with central composite as statistical design in order to attain high xylose selectivity.

2. Methods

2.1. Raw material

Oil palm empty fruit bunch (OPEFB) fiber was collected from local palm oil mill (United Oil Palm Industries Sdn Bhd, Malaysia), sun-dried and ground to a particle size <1 mm. The homogenized OPEFB biomass was then oven-dried at 105 °C for overnight and was analyzed following standard method for determination of its main composition (Garrote et al., 1999).

2.2. Acid hydrolysis

Acid hydrolysis of OPEFB biomass were carried out in 125 ml Erlenmeyer flasks. The media consisted of 2–6 g H_2SO_4/100 g liquor using a charge of 1 g OPEFB fiber/8 g liquor on dry basis. Operating temperature of hydrolysis were varied between 100 and 130 °C and samples were collected at various time intervals in the range of 30–90 min. After reaction was completed, solids were separated from aqueous solution by filtration. The filtrate was analyzed for xylose, glucose, acetic acid and furfural.

2.3. Analytical methods

Xylose and glucose in the acid hydrolysate were analyzed by High Performance Liquid Chromatograph (HPLC, SHIMADZU) using SUPELCOSIL LC-NH_2 column and RI detector. Aqueous acetonitrile (75%) was used as mobile phase with flow rate of 1.5 ml/min and oven temperature was maintained at 50 °C. Acetic acid and furfural were analyzed by Gas Liquid Chromatograph (GLC, Perkin Elmer) using 80/120 carbopack column with nitrogen as carrier gas and detection was done by FID. The column and injector temperature was maintained at 225 and 250 °C, respectively while oven was operated at 175 °C.

2.4. Experimental design and RSM

In the experimental plan, response surface methodology (RSM) was utilized to optimize the hydrolysis process and a 2^3 rotatable central composite design (CCD) was adopted in order to fit a second order model and the design consisted of 20 sets of experiments. The basic theoretical aspects, the fundamental assumptions and the experimental implications of RSM have been discussed elsewhere (Montgomery, 2001). The second order model was selected for predicting the optimal point and is expressed as

$$Y = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 + \beta_{11} X_1^2 + \beta_{22} X_2^2 + \beta_{33} X_3^2 + \beta_{12} X_1 X_2 + \beta_{13} X_1 X_3 + \beta_{23} X_2 X_3 \quad (1)$$

where Y represents response variable (xylose yield and selectivity), β_0 is interception coefficient, β_1 and β_2 are linear terms, β_{11}, β_{22} and β_{33} are quadratic terms and X_1, X_2 and X_3 are independent variables studied (temperature, reaction time and acid concentration). Regression analysis was performed by Design Expert v.6.0.7 (Stat-Ease Inc. Minneapolis). Fischer's test was used for determination of type of model equation, while the student's t-test was performed for determination of statistical significance of regression coefficients.

3. Results and discussion

3.1. Composition of empty fruit bunch

Analysis of OPEFB was carried out for determination of principal components using quantitative acid hydrolysis. The composition is shown in Table 1, expressed on an

Table 1
Main components of oil palm empty fruit bunch fiber (on an oven-dry basis)

Main fraction	Composition (%)
Glucan	42.85
Xylan	24.01
Lignin (acid insoluble)	11.70
Ash	0.52
Others	20.92