ENHANCING XYLOSE EXTRACTION FROM PASSION FRUIT PEEL THROUGH OPTIMAL HYDROLYSIS

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ABSTRACT

Brazil is one of the main highlights in the global agroindustry, and this position extends to several areas, including the production of passion fruit, used mainly in the preparation of juices and their derivatives, resulting in large amounts of waste arising from its processing. Therefore, the purpose of the present work was to promote the use of passion fruit peel (PFP) from acid hydrolysis, considering the variables of acid concentration and pre-treatment time to maximize the release of xylose and obtain xylitol through fermentation. A factorial experimental design was carried out to analyze the effect of H_2SO_4 concentration (0.03M; 0.2M; 0.6M; 1M; 1.17M) and time (5.9 min.; 10 min.; 20 min.; 30 min.; 34.1 min.) in the yield of released sugars and in improving the accessibility to cellulose and hemicellulose. Subsequently, the efficiency of pretreated hydrolysis was evaluated regarding the production of fermentable sugars, especially xylose. The resulting products were analyzed using high-performance liquid chromatography (HPLC), and the evaluation of morphology and structural changes was carried out using a scanning electron microscope (SEM). The highest glucose yield from PFP biomass was recorded from pretreated hydrolysis at 0.71 M H_2SO_4 concentration in 21.84 minutes. From the results obtained, it was concluded that PFP has significant potential for the biotechnological production of xylose through acid hydrolysis carried out at different acid concentrations and heat treatment times, making it possible to add value to the residue from the obtaining sugar destined for microbial fermentation and xylitol production.

Keywords: Biotechnological valorization. Sustainability. Acid hydrolysis. Fermentable sugars. Xylitol.

1 INTRODUCTION

The Brazilian agro-industry, especially the fruit production sector, is vital for the national economy, serving both the domestic market and exports. Among the various products exported by Brazil, passion fruit stands out due to its high production, low seasonality, and promising acceptance by the public in various countries, mainly the Netherlands, the USA, and the UK (which buy more than 90% of the fruit exported) (EMBRAPA, 2022). Approximately 697,000 tons of this fruit were produced in 2022, making the country the world's largest producer, with the states of Bahia (228 t/2022) and Ceará (148 t/2022) standing out, whose combined production corresponds to around 54% of national production (IBGE, 2022). However, the passion fruit processing industry generates significant amounts of residues from its processing, especially passion fruit peels (PFP), which represent around 54,000 tons of discards every year (ZHAO et al., 2023)

A promising alternative for reducing the environmental impacts caused by the large volume of agro-industrial residues is to valorize these biomasses. Using technologies that use them as raw materials, it is possible to obtain compounds with more excellent added value, thus aligning with a green economy. In a biorefinery context, in which the full use of the lignocellulosic fractions of biomass is pursued to obtain various products, research has aimed to develop technologies to produce intermediates and products from these underutilized residues. It also contributes to promoting the circular economy, closing the loop of industrial processes, resulting in efficient waste management, and creating the opportunity to effectively balance environmental and economic aspects, allowing remarkable advances in the scientific and technological spheres.

Several reports show the use of PFP to obtain pectin and nutritional flours. Still, it also has the potential to be used as a raw material to generate products with higher added value. PFP is composed mainly of pectin, cellulose, hemicellulose, and lignin, which, after acid hydrolysis, can lead to fermentable sugars such as glucose and xylose. The last sugar is one of the main components of hemicellulose. Microorganisms can metabolize and convert it into xylitol, a polyol with high sweetening power and anti-cariogenic action widely used in the food, pharmaceutical, and dental industries (ALBUQUERQUE, 2015).

In this context, the purpose of this study was to improve the process of hydrolysis of passion fruit peel (PFP) using a rotational central composite design (RCCD) methodology, considering the variables of acid concentration and pre-treatment time to maximize the release of xylose to obtain xylitol by fermentation.

2 MATERIAL & METHODS

2.1 Obtention and production of yellow passion fruit peel flour

The experiments used the epicarp and mesocarp of yellow passion fruit (*Passiflora edulis flavicarpa*) obtained from a local market in Fortaleza, Ceará, Brazil. The peels were washed three times, dried at 60 °C, crushed to obtain flour, and standardized using sieves with a mesh size of approximately 0.84 mm (20 mesh).

2.2 PFP hydrolysis

The influence of the H_2SO_4 concentration and the of heat treatment at 121 °C duration on the release of fermentable sugars was studied using a rotating central composite experimental design (RCCD). The independent factors evaluated were H_2SO_4 concentration (0.03M; 0.2M; 0.6M; 1M; 1.17M) and time (5.9 min; 10 min; 20 min; 30 min; 34.1 min). The biomass proportion of passion fruit flour was standardized at 10% (w/v). After hydrolysis, the solid fraction was removed by vacuum filtration, and the liquid fraction was analyzed for the content of sugars released.

2.3 Analytical methods

2.3.1 Carbohydrates concentration

The quantification of xylose and glucose concentrations was determined using High-Performance Liquid Chromatography (HPLC) (Waters, Milford, MA, USA), equipped with a Waters refractive index detector (Model 2414) and a Supelco 610-H column, maintained at a temperature of 30 °C. The eluent consisted of phosphoric acid (H_3PO_4) at a concentration of 0.1% (w/v) and a flow rate of 0.5 mL/min with a sample injection volume of 20 µL.

2.3.2 Morphological evaluation

The morphology and structural changes were analyzed using images from a scanning electron microscope (FEG Quanta 450) with energy-dispersive X-ray spectroscopy (EDS). The samples were previously placed on carbon tape and then coated with silver using a Quorum QT150ES metallizer. The SEM camera was subjected to a pressure of 10 Pascal, with an incident electron beam of 20 kV.

2.3.3 Experimental design analysis

The Statistica v7.0 software (Statsoft) was used to prepare and analyze the experimental designs. The response surface methodology (RSM) was applied to analyze the results. The statistical evaluation of significance was carried out using a one-factor analysis of variance (ANOVA) with a significance level of 95%.

3 RESULTS & DISCUSSION

The results for xylose and glucose production from the conducted experimental design are presented in Table 1. It is possible to observe that the points with the maximum yield of both sugars were at the central points, with an average of 18.58 g/L for xylose and 9.68 g/L for glucose. On the other hand, the condition that led to the lowest release of both sugars was at the acid concentration of 0.03 M per minute, demonstrating the significant influence of acid concentration on the hydrolysis process of cellulose and hemicellulose fractions. It is also noticeable that at acid concentrations above 1 M, the effect on sugar release was unfavorable, resulting in reduced sugar concentrations. This is because, at higher temperatures, sulfuric acid can also hydrolyze polysaccharides into monosaccharides. The monosaccharides are unstable at low pH and high temperatures, leading to an additional reaction to form formic acid via furan intermediates (YOON; HAN; SHIN, 2014).

Table 1. Experimental design and response for dilute acid hydrolysis of passion fruit peel using H_2SO_4

Run	H_2SO_2 (M)	Time (min)	Xylose (g/L)	Glucose (g/L)
1	0.20	10.00	2.96 ± 0.09	1.27 ± 0.66
2	0.20	30.00	4.80 ± 0.05	3.12 ± 0.01
3	1.00	10.00	7.50 ± 0.43	3.24 ± 0.58
4	1.00	30.00	10.90 ± 0.31	6.57 ± 0.05
5	0.03	20.00	1.36 ± 0.10	1.30 ± 0.05
6	1.17	20.00	11.89 ± 0,02	3.88 ± 0.04
7	0.60	5,9.00	3.49 ± 0,01	2.26 ± 0.95
8	0.60	34,10	10.95 ± 0,41	6.17 ± 0.34
9	0.60	20.00	18.20 ± 0.11	9.20 ± 074
10	0.60	20.00	19.03 ± 0.01	9.98 ± 1.02
11	0.60	20.00	18.50 ± 0.05	9.85 ± 0.90

The purpose of this research was to analyze the impact of acid concentration and hydrolysis time on the production of xylose and glucose. The study analyzed two charts, one of which was the Pareto chart (Figure 1), which illustrates the influence of both variables. investigate the effects of acid concentration and hydrolysis time on the production of xylose and glucose. The Pareto chart, which displays the influence of both acid concentration and hydrolysis time, is presented in Figure 1.

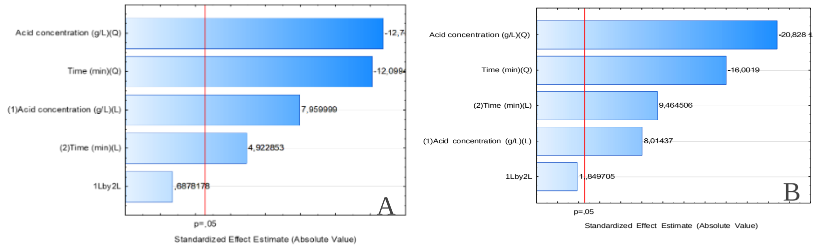

Figure 1. Pareto Chart for xylose (A) and glucose (B) release in the hydrolysis of passion fruit peel under H_2SO_4 treatment.

Meanwhile, Figure 2 shows the response surface, highlighting the optimal region for obtaining xylose and glucose. The F-test and ANOVA analysis were used as criteria to determine the significance of the fitted models. At a 95% confidence interval, all models demonstrated statistical significance, as the calculated F-values were significantly higher than the tabulated values (61.68 for Equation 1 and 154.41 for Equation 2, compared to F5,10= 3.33 and F5,5= 5.05, respectively). Additionally, satisfactory correlation coefficients were obtained (R^2= 0.96 for Equation 1 and R^2= 0.98 for Equation 2).

$$Xylose\ (g/L) = -25.0433+49.7086x+2.4011y-36.4212x2+0.0975xy-0.0566y2\ (R2 = 0.96)\ (1)$$

$$Glucose\ (g/L) = -12{,}2562+26{,}9448x+1{,}1663y-21{,}6448x2+0{,}0925xy-0{,}0272y2\ (R2 = 098)\ (2)$$

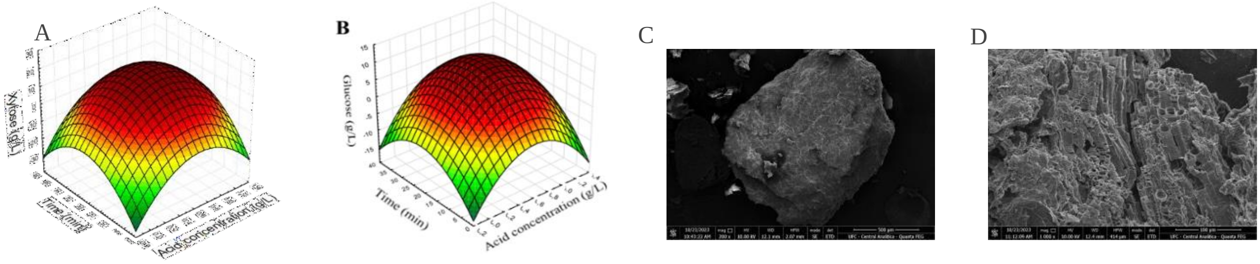

Figure 2. Surface response to the release of xylose (A) and glucose (B) in the hydrolysis of passion fruit peel under treatment with H_2SO_4 and morphological structure before hydrolysis (C) and after hydrolysis in the condition with higher xylose release, 0.6 M of H_2SO_4 for 20 min (D).

Based on the study conducted, it was found that the optimal concentration of H_2SO_4 was 0.71 M, and the ideal time duration was 21.84 minutes for the maximum release of xylose from PFP biomass. The concentration levels that exceeded 18 g/L were obtained under these optimal conditions. Therefore, these conditions are perfect for obtaining hydrolysate for xylitol production through fermentation. Additionally, Figures 2C and 2D display a noticeable change in the PFP structure morphology, indicating the emergence of crystalline cellulose, which is more robust than amorphous cellulose. This indicates that the treatment was effective in breaking down hemicellulose into xylose.

4 CONCLUSION

Through experimental design, it was possible to optimize the production of xylose from the acidic hydrolysis of passion fruit peel. The maximum concentration of xylose achieved was 19.03 g/L by adjusting the variables of acid concentration and thermal treatment time. These conditions can be used to obtain the sugar for microbial fermentation and xylitol production. In conclusion, the optimization of xylose production from passion fruit peel can be achieved through specific adjustments of acid concentration and thermal treatment time variables.

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