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## Determination of optimum conditions of acid hydrolysis of the fiber of oil palm (*Elaeis guineensis*) in order to obtain xylose utilization in bioprocess

B.M.Radomski<sup>1</sup>, W.A.Gonzalez<sup>1</sup>, S.B.Fiaux<sup>2</sup>

<sup>1</sup>Instituto Militar de Engenharia - Depto de Química CEP: 22290-270 - Urca-RJ, (BRAZIL)

<sup>2</sup>Universidade Federal Fluminense – Faculdade de Farmácia CEP: 24241-000 - Niteroi-RJ, (BRAZIL)

E-mail : biancaradomski@yahoo.com.br; sorele@vm.uff.br; d5wilma@gmail.com

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### ABSTRACT

The palm fiber, lignocellulosic material, rich in fermentable sugars have potential for use in different bioprocesses. The acid hydrolysis process is commonly employed for the fractionation of these materials for the use of biotechnology xylose contained in hemicellulosic hydrolysates. This study aimed to produce xylose from the residue of the biodiesel industry through hydrolysis using sulfuric acid, aiming particularly bioethanol use in bioprocesses. For optimization of acid hydrolysis, there was an experimental design, independent variables were the acid concentration, temperature and reaction time on the response variable concentration of xylose released. The maximum concentration of xylose was obtained  $49.46 \times 10^2$  kg / m<sup>3</sup>, by hydrolysis using 0.46 M sulfuric acid, at 120 °C for 30 minutes.

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### KEYWORDS

Palm fiber;  
Experimental design,  
Xylose.

### INTRODUCTION

Lignocellulosic raw materials are renewable resources abundantly found in nature. These are sources of hexoses and pentoses with potential use for the production of fuel ethanol, chemicals and food products<sup>[1]</sup>.

The use of biomass waste produced in the country still requires accurate assessments of their potential recovery economically viable and comprehensive analysis of their life cycles and energy products. Despite the limited information available on these parameters, studies in this regard are supported by the National Energy Plan 2030<sup>[7]</sup> for the collection of information more con-

sistently, compared to expectations of appreciation of these wastes for various applications sustainable.

In the study presented here, there is the residue of biodiesel production from palm oil, composed of fibers, shells and empty bunches, which currently often used as boiler fuel or as fertilizer. The lignocellulosic material can be a prime source of raw materials for bioprocesses, and for the production of second generation biofuels and chemicals, and a way to add value to the production chain of biodiesel from palm oil.

The objective of this study was to determine the conditions of hydrolysis using sulfuric acid to release xylose from the cluster of palm fibers, lignocellulosic material.

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## MATERIALS AND METHODS

The fiber of the bunch of palm oil has been received from the Experimental Field of Embrapa in Urubu, Ceru, Manaus. Initially, the material (about 10.00 grams) was ground in a blender to pass through 16 mesh sieve and placed in a beaker, which was submitted to the autoclave at 121 ° C for 1 hour. This procedure was to reduce the size of the particles and clean the raw material, making the cellular structure more accessible to chemical treatment<sup>[4]</sup>.

For the hydrolysis of palm fiber, we used sulfuric acid 98% PA, acid selected in preliminary experiments<sup>[9]</sup>. The reaction conditions were selected from a full factorial experimental design for the following variables and domains: concentration of sulfuric acid - 0.05 to 0.46 M; hydrolysis time - 10 to 30 minutes, temperature of hydrolysis - 120 to 190 ° C . The variables and their limits are chosen based on the literature on acid hydrolysis of lignocellulosic materials<sup>[3,6,10,13]</sup>.

Variables fiber mass and volume of acid were kept constant and equal to 1.00 gram and 50.00 ml, respectively. The concentration of xylose released was the response variable. Two replicas of the central point were added to the plan to verify the reproducibility and the possibility of non-linearity (curvature) in the range studied. The plan is presented in TABLE 1. The statistical analysis was performed using Statistica software version 5.5 from Statsoft.

Acid hydrolysis of fibers was performed in 300 mL Parr reactor closed, using a thermocouple and stirring of 30 rpm, constant in all experiments. The pressure inside the reactor PARR is related to the temperature of each experiment and the pressure value is read by a manometer attached.

After hydrolysis, the reactor content was filtered and frozen for later analysis of xylose. (The analysis of xylose was performed by the phloroglucinol<sup>[11,12]</sup> and spectrophotometer at a wavelength of 540 nm.

## RESULTS AND DISCUSSION

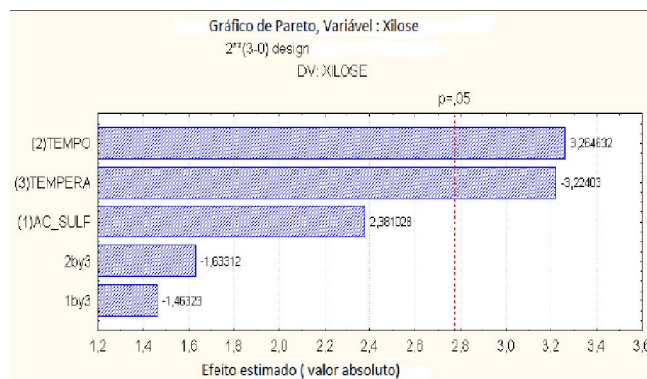
The experimental design and results are presented in TABLE 1.

Figure 1 shows the Pareto chart obtained from the statistical analysis of the experimental design. In this

chart, you can check the variables that influence the concentration of xylose released. Those whose bars extend beyond the vertical line (p-level 0.5) have significant influence on the response variable with 95% confidence<sup>[8]</sup>.

**TABLE 1 : Experimental conditions and the response variable (concentration of xylose released) experimental design used to determine the conditions of acid hydrolysis of palm fiber.**

Experiment	Acid concentration (M)	Time (minutes)	Temperature (°C)	xylose (10 <sup>2</sup> Kg/m <sup>3</sup> )
1	0,05	10	120	8,55
2	0,05	10	190	12,10
3	0,05	30	120	35,02
4	0,05	30	190	14,99
5	0,46	10	120	30,09
6	0,46	10	190	11,65
7	0,46	30	120	49,46
8	0,46	30	190	24,03
9 (C)	0,25	20	135	13,79
10 (C)	0,25	20	135	13,97



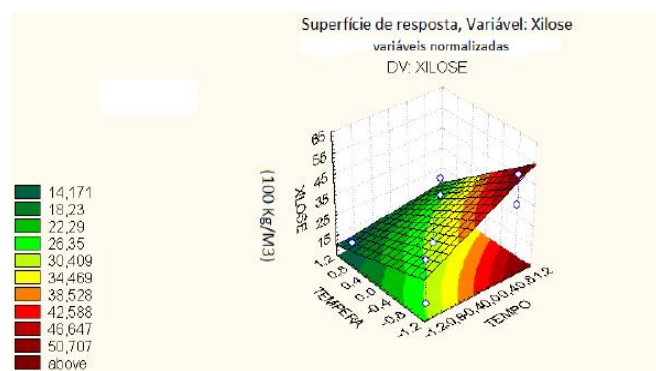
**Figure 1 : Pareto chart for the concentration of xylose as the response variable.**

You can see that the main effects of the variables of time and sulfuric acid contributes to the increased concentration of xylose (positive sign in the bar). The main effect of temperature is also significant for the response variable, but its increase leads to less concentration of xylose released (negative sign in the bar). The interaction between temperature and time appears to be significant, verifying that the response will be maximum if the weather is at its upper level and the temperature at its lowest level. The curvature was not significant in the model.

The time and temperature are the most influential variables on the response. This can also be seen in

TABLE 1, noting that for the same time and acid concentration in a temperature increase implies a decrease in the concentration of xylose obtained, which may have been a result of the degradation of this compound furfural<sup>[5]</sup>. The opposite is observed in experiments 1 and 2. Probably the first experiment, the low values ?? of concentration of sulfuric acid residence time and temperature of hydrolysis was insufficient to release a higher amount of xylose from lignocellulosic material.

Figure 2 shows the response surface as a function of temperature and time generated in the analysis of planning. The graph can be observed that the concentration of xylose increased with increasing hydrolysis time and decreases with increasing temperature.



**Figure 2 : Response surface as a function of temperature and time of hydrolysis (acid concentration 0.46 M).**

The equation generated by the model is presented below. The variables are standardized, ie, represented by +1 and -1 its maximum to minimum.

$$\text{Xylose released (10}^2 \text{ Kg/m}^3) = 21,365 + 5,571 * [\text{acid}] + 7,639 * t - 7,544 * T - 3,821 * [T] * t - 3,424 * [\text{acid}] * T,$$

**Where:**

**[acid]:** concentration of sulfuric acid;

**t:** hydrolysis time;

**T:** temperature of hydrolysis

**Coefficient of determination (R<sup>2</sup>) = 0,89; Adjustment = 0,75**

By the equation at the point indicated as optimal within the domain, the concentration of acid should be +1 (0.46 M), time +1 (30 minutes) -1 and temperature (120 ° C). Calculating the concentration of xylose released with these values, we 49,36\*10<sup>2</sup> Kg/ m<sup>3</sup>, value close to that seen in experiment 7, taken under the same conditions used in the calculation.

## CONCLUSIONS

Based on the experimental design performed in order to enhance the release of xylose by acid hydrolysis of palm fiber, it was concluded that the main effect of all variables (time, acid concentration and temperature) and time-temperature interaction are significant on the concentration of xylose. Within the domain studied the hydrolysis conditions that lead to greater response are the concentration of sulfuric acid 0.46 M, time 30 minutes and temperature of 120°C. An effective treatment of palm fibers should lead to high release of xylose, with minimal degradation. The xylose is a substrate to be used in bioethanol production, final goal of the project. Degradation can lead to production of fermentation inhibitors such as furfural and needs to be removed<sup>[2]</sup>. The experimental design allowed us to check the condition of hydrolysis to obtain the largest concentration of xylose, ie, its biggest release of these fibers with less degradation. In later work, the xylose is used to produce second generation biofuels.

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