Effect of the hydrochloric acid concentration on the hydrolysis of sorghum straw at atmospheric pressure

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Abstract

Sorghum straw is a raw material useful for the xylose production by hydrolysis. The main application of xylose is its bioconversion to xylitol, a functional sweetener with important technological properties. The objective of this work was to study the hydrolysis of sorghum straw with hydrochloric acid at 100 °C. Several concentrations of HCl (2–6%) and reaction time (0–300 min) were evaluated. Kinetic parameters of mathematical models for predicting the concentration of xylose, glucose, acetic acid and furfural in the hydrolysates were found and used to optimise the process and compared with results reported in the literature using other conditions and acids. Optimal conditions found for hydrolysis were 6% HCl at 100 °C for 83 min, which yielded a solution with 21.3 g xylose/l, 4.7 g glucose/l, 0.8 g furfural/l and 2.8 g acetic acid/l.
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1. Introduction

Sorghum straw is commonly used as livestock feed. The hydrolysis of sorghum straw to produce xylose solutions could generate an added value for this renewable, cheap, widely available and abundant resource. The hydrolysis of sorghum straw also eliminates a waste when the straw is left unused.

Among the treatments evaluated, dilute acid hydrolysis appears to be in the best position from the economic viewpoint (Wyman, 1994). In a single step, this technology causes several alterations in the raw materials, including hemicellulosic sugars release, mainly xylose (Parajó, Santos, & del Río, 1995a, 1995b). The main application of xylose is its bioconversion to xylitol, a functional sweetener with important technological properties like anticarcinogenicity, low caloric value and negative heat of dissolution (Kim, Ryu, & Seo, 1999; Parajó, Santos, & Vázquez, 1997).

The hydrolysis reactions in dilute-acid medium are very complex, mainly because the substrate is in a solid phase and the catalyst in a liquid phase. The mechanism for this hydrolysis reaction has been reported by Herrera, Téllez-Luis, Ramírez, and Vázquez (2003). Empirical models are commonly used due to the difficult modelling of these processes. Dilute acids lead to a limited hydrolysis called prehydrolysis. This consists in the hydrolysis of the hemicellulosic fraction, leaving the cellulose and lignin fractions almost unaltered. Hydrochloric (Herrera et al., 2003) and sulphuric acids (Téllez-Luis, Ramírez, & Vázquez, 2002a, 2002b) have been used to catalyst the hydrolysis of sorghum straw. For other raw materials hydrofluoric (Franz, Erckel, Riehm, Woernle, & Deger, 1982) and acetic (Conner & Lorenz, 1986) acids were also employed as catalysts.

The product of the hydrolysis is a solution containing mainly sugars such as xylose, glucose and arabinose. Other compounds such as oligomers, furfural and acetic acid are also released. Bonds in the hemicellulosic fraction are weaker than in the cellulosic fraction. Therefore, using selected operational conditions, it is possible to hydrolyse almost quantitatively the hemicelluloses

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leaving the cellulose and lignin in the solid residue, which can be processed for the production of lactic acid, ethanol, single cell protein through saccharification–fermentation or for the production of paper pulp (Caraschi, Campana, & Curvelo, 1996; David, Fornasier, Greindl-Fallon, & Vanlautem, 1985; Grethlein & Converse, 1991; Parajó, Santos, Domínguez, Vázquez, & Alvarez, 1995). The economic interest in biotechnological production of xylitol can be enhanced if the carbon source needed, xylose, could be obtained from the hydrolysis of a low-cost lignocellulosic waste such as sorghum straw. For this application, hydrolysates with high concentration of xylose and low concentration of growth inhibitors (furfural and acetic acid) are desired. The effect of acetic acid on the growth of microorganisms is not clear and depends on the strain. Ferrari, Neirotti, Albornoz, and Saucedo (1992) reported that 10.5 g acetic acid/l hindered the growth of *Pichia stipitis*, but Palmqvist, Almeida, and Hahn-Hägerdal (1999) found that 9–10 g acetic acid/l enhanced the growth and productivity of *Saccharomyces cerevisiae*.

There are only a few studies on the hydrolysis of sorghum straw. Only the sulphuric-acid hydrolysis at atmospheric and autoclave pressure (Téllez-Luis et al., 2002a, 2002b) and the hydrochloric-acid hydrolysis at autoclave pressure (Herrera et al., 2003) have been studied. The objective of this work was to study the hydrolysis of sorghum straw at 100 °C (atmospheric pressure) using dilute hydrochloric acid. Kinetic parameters for the mathematical models predicting the concentration of xylose, glucose, acetic acid and furfural in the hydrolysates were estimated and used to optimise the process.

2. Material and methods

2.1. Materials

Sorghum bicolor straw collected from a local farm (Tamaulipas, Mexico) was used as raw material. The straw was air-dried, milled, screened to select the fraction of particles with a size lower than 0.5 mm, homogenized into a single lot and stored until needed.

2.2. Analytical methods

Analyses of the main fractions (cellulose, hemicelluloses and Klason lignin) were carried out using a quantitative acid hydrolysis under standard conditions (Garrote, Domínguez, & Parajó, 1999). The main composition of the sorghum straw was: cellulose, $34 \pm 1\%$; xylan, $20 \pm 1\%$, hemicelluloses, $24 \pm 1\%$ and lignin, $20 \pm 1\%$.

Treatments were performed at 100 °C in media containing 2, 4 or 6 g HCl/100 g liquor using a charge of 1 g

straw/10 g liquor. At 20, 40, 60, 180 and 300 min, liquor samples were taken from the reaction media and analysed. The samples were diluted with water (1/10 v/v), centrifuged to separate the insolubilized phenolic fraction and the supernatant used to determine the composition of the hydrolysates. The supernatant was diluted with water (1:10 v/v) and analysed by HPLC for glucose, xylose, arabinose and acetic acid. The HPLC analyses were carried out using a Transgenomic ION-300 column (oven temperature = 45 °C) with isocratic elution (flow rate = 0.4 ml/min; mobile phase: H_2SO_4 0.0025 M) and a refraction index detector. Furfural was analysed by UV–Vis spectroscopy at 280 nm.

2.3. Statistical analysis

All determinations were carried out using three samples and the related data were expressed as averages values. Non-linear regression analyses of experimental data were performed with a commercial optimisation routine based on the Newton's method (Solver, Microsoft Excel 2000, Microsoft Corporation, Redmont, WA, USA), by minimizing the sum of the squares of deviations between experimental and calculated values as previously described (Ramírez, Santos, Morales, Morrissey, & Vázquez, 2000).

2.4. Kinetic models

Due to the difficulty to find a strict mechanism for hydrolysis reactions, it is usual to use simplified models to determine the kinetics of the hydrolysis of lignocellulosic materials. The models proposed in the literature use pseudo-homogeneous irreversible first-order reactions. The first model used successful was proposed by Saeman (1945):

$$\text{Cellulose} \xrightarrow{k_1} \text{Glucose} \xrightarrow{k_2} \text{Decomposition products} \quad (1)$$
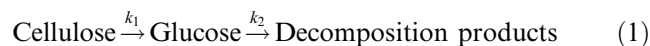
This model was designed for the hydrolysis of cellulose from fir wood using sulphuric acid. The model was also applied to the hydrolysis of the hemicellulosic fraction (Grant, Han, Anderson, & Frey, 1977; Téllez-Luis et al., 2002a, 2002b) and could be used for hemicellulosic hydrolysis as follows:

$$\text{Xylan} \xrightarrow{k_1} \text{Xylose} \xrightarrow{k_2} \text{Decomposition products} \quad (2)$$
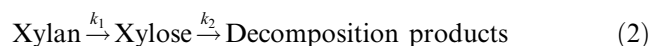
where k_1 is the rate of xylose release (min^{-1}) and k_2 is the rate of xylose decomposition (min^{-1}). Based on this reaction model and solving differential equations leads to Eq. (3), which expresses xylose concentration $[X]$ as a function of time (t):

$$[X] = \frac{k_1[Xn_0]}{k_2 - k_1}(e^{-k_1 t} - e^{-k_2 t}) + [X_0]e^{-k_2 t} \quad (3)$$

The initial xylan and xylose concentrations are defined at time 0 as $[Xn_0]$ and $[X_0]$, respectively. Assuming that $[X_0]$ is close to 0, a simplification of the model yields:

$$[X] = \frac{k_1[Xn_0]}{k_2 - k_1}(e^{-k_1 t} - e^{-k_2 t}) \qquad (4)$$

A similar model can be developed to describe the increase in glucose concentration, but in this case the decomposition reactions were negligible and the model can be simplified as follows:

$$\text{Glucan} \xrightarrow{k_3} \text{Glucose} \qquad (5)$$

where k_3 is the rate of glucose release (min^{-1}). Solving differential equations leads to Eq. (6), which expresses the glucose concentration $[G]$ as a function of time (t):

$$[G] = [G_0] \cdot (1 - e^{-k_3 t}) \qquad (6)$$

where $[G_0]$ is the potential glucose concentration which was estimated by regression.

Furfural is the main degradation product in the hydrolysis of lignocellulosic materials to obtain xylose. A model has also been developed for furfural concentration. In this case, the rate for furfural generation is k_4 (min^{-1}). The equation that expresses furfural concentration $[F]$ as function of time is

$$[F] = [F_0] \cdot (1 - e^{-k_4 t}) \qquad (7)$$

where $[F_0]$ is the potential concentration of furfural (g/l) that was determined by regression analysis.

Acetic acid derives from the hydrolysis of the acetyl groups bound to the hemicellulosic monomers. The modelling of acetic acid can be developed on the basis of the equation:

$$\text{Acetyl groups} \xrightarrow{k_5} \text{Acetic acid} \qquad (8)$$
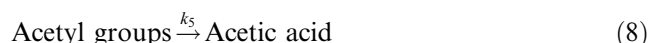
where the regression parameter k_5 is the rate of its generation (min^{-1}).

Acetic acid concentration (Ac) can be expressed as a function of time (t) as

$$Ac = Ac_0 \cdot (1 - e^{-k_4 \cdot t}) \qquad (9)$$

where the regression parameter Ac_0 is the potential concentration of acetic acid and k_4 the rate of acetic acid generation (min^{-1}).

Eqs. (4), (6), (7) and (9) have been applied to model the hydrolysis of sorghum straw with hydrochloric acid. Non-linear regression analyses were performed to obtain the kinetic parameters and constants. The results were statistically evaluated with the coefficient of determination r^2 and F-test probability to establish the reliability of the models.

3. Results and discussion

The high xylan content ($20 \pm 1\%$) makes the sorghum straw adequate for xylose production. The potential concentration of xylose corresponds to the quantitative conversion of xylan to xylose. Through material balance, the potential concentration was 22.3 g xylose/l liquor.

Hydrolysates were obtained using HCl at 100 °C. The range of HCl concentration (2–6%) were selected according to the values reported for dilute acid hydrolysis of lignocellulosic materials (Aguilar, Ramírez, Garrote, & Vázquez, 2002; Bustos, Ramírez, Garrote, & Vázquez, 2002). The temperature studied (100 °C) was selected because data at 122 °C previously reported suggested that a lower temperature could enhance hydrolysis. Thus, the results obtained in this work will be compared with data reported using 122 °C (Herrera et al., 2003).

Fig. 1(a) shows the concentration of xylose, glucose, furfural and acetic acid released during the hydrolysis of sorghum straw performed with 2% HCl at 100 °C. Sugar concentrations increased progressively up to 17.3 g xylose/l and 3.8 g glucose/l at 300 min while decomposition products increased only up to 1.0 g furfural/l and 2.5 g acetic acid/l. The maximum concentration of xylose obtained was higher at 100 °C than at 122 °C due to the undesired secondary reactions responsible of xylose decomposition was slower at 100 °C than at 122 °C. Moreover, a temperature of 100 °C seems enough to hydrolysis the xylan of sorghum straw.

Fig. 1(b) shows the concentration of xylose, glucose, furfural and acetic acid released during the hydrolysis of sorghum straw at 100 °C with 4% HCl. Xylose concentration increased up to 19.9 g/l at 300 min. The best result using 4% HCl at 122 °C was 15.1 g xylose/l at 60 min. This suggests that the increase of catalyst concentration from 2% to 4% at 100 °C improves the hydrolysis reaction and do not increase the rate of decomposition reactions as it was observed at 122 °C. Glucose concentration grew more at 100 °C than 122 °C (5.1 vs 4.9 g/l) but it was not significant. The comparison of the decomposition products gave results dissimilar. Furfural concentration was lower at 100 °C than at 122 °C (1.1 vs 3.3 g/l) while acetic acid concentration was higher at 100 °C than at 122 °C (3.6 vs 1.6 g/l).

Fig. 1(c) shows the concentration of products released during the hydrolysis of sorghum straw at 100 °C with 6% HCl. Xylose concentration had a maximum of 19.7 g/l at 180 min and then decreased with a slight slope. This result compare very well with the maximum value obtained using 122 °C, 16.0 g/l. The xylose concentration obtained at 100 °C was 88.3% of the potential concentration. Glucose concentration reached up to 5.3 g/l at 180 min. Furfural and acetic acid also increased up to 1.7 and 3.6 g/l, respectively. Furfural was 2.8 times
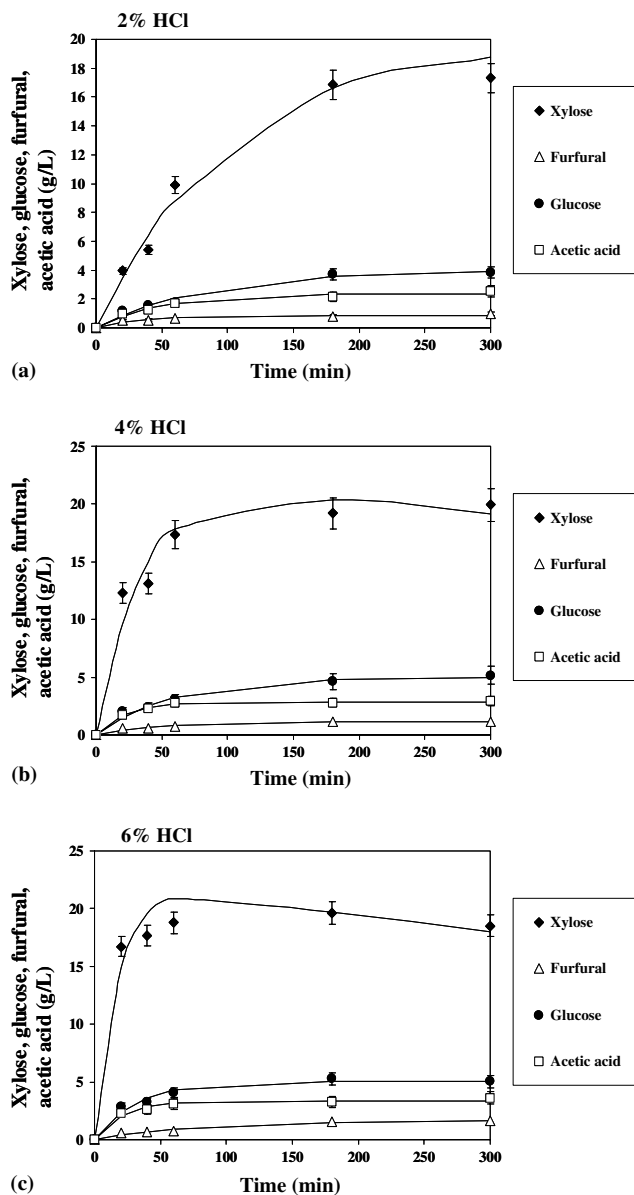
Fig. 1. Experimental and predicted dependence of the products generated during the hydrolysis at 2%, 4% or 6% HCl at 100 °C and different time.

Table 1
Kinetic and statistical parameters of products released during the HCl hydrolysis of sorghum straw at 100 °C

	2% HCl	4% HCl	6% HCl
Xylose			
k_1 (min^{-1})	0.00852	0.02861	0.05649
k_2 (min^{-1})	0.00043	0.00058	0.00076
r^2	0.9842	0.9571	0.9663
F-prob	0.9305	0.9276	0.9262
Glucose			
k_3 (min^{-1})	0.01198	0.01788	0.03097
$[G_0]$	4.02	4.98	5.08
r^2	0.9807	0.9782	0.9747
F-prob	0.9719	0.9546	0.9742
Furfural			
k_4 (min^{-1})	0.02802	0.02023	0.01258
$[F_0]$	0.87	1.14	1.66
r^2	0.9147	0.9332	0.9685
F-prob	0.9934	0.9543	0.9783
Acetic acid			
k_5	0.02072	0.04344	0.04817
$[Ac_0]$	2.38	2.89	3.36
r^2	0.9764	0.9951	0.9765
F-prob	0.9842	0.9952	0.9968

at 22.3 g/l, taking into account the solid/liquid ratio and the stoichiometric ratios. Table 1 shows the kinetic and statistical parameters obtained for xylose released during the hydrolysis of sorghum straw at 100 °C.

Fig. 1 show the experimental and predicted data for these hydrolyses at each concentration of HCl. Comparing the values of k_1 and k_2, the reactions of xylose generation appear to be accelerated with increasing HCl concentration more than the reaction of decomposition. Although the temperature is lower, the values of k_1 are higher than using 122 °C (Herrera et al., 2003). This could be explained because the substrate is in a solid phase and the catalyst in a liquid phase. The high temperature can modify the structure of the lignocellulosic material making difficult the access of the proton to the raw material due to the hydrophobicity of the lignin. However, the decomposition reactions of xylose take place only in the liquid phase, being lower the values of k_2 at 100 °C than at 122 °C as expected.

The determination coefficient (r^2) showed a good agreement between experimental and predicted data for all regressions (Table 2). The value of F-test probability showed that the models are accurate for the range of acid concentration studied.

Optimizing the models for separate, they predict that the maximum xylose concentration can be obtained using 6% HCl concentration at 100 °C during 77 min, being 21 g xylose/l. However, a generalized model for predicting at all HCl concentration in the range 2–6% can be developed. Kinetic parameters were correlated with the HCl concentration (C_a) by means of the empirical equation:

lower than at 122 °C (4.7 g/l) and acetic acid was 2.1 times higher (1.6 g/l at 122 °C). This results suggest that it is better to use 100 °C than 122 °C due to the high concentration of xylose and the low concentration of furfural obtained at 100 °C. Although the acetic acid concentration was increased at 100 °C, this compound is less toxic than furfural, which was decreased at 100 °C.

3.1. Kinetic modelling of xylose concentration

The xylose concentration in the hydrolysis of sorghum straw with hydrochloric acid at 100 °C has been modelled using Eq. (4). Xylan concentration, $[Xn_0]$, was fixed

$$k_j = k_0 C_a^n \qquad (10)$$

where j is an integer in the range 1–5, k_0 and n are regression parameters and C_a is the acid concentration expressed as % (w/w). In the xylose model, k_1 correlated with HCl concentration as shown in Eq. (11) of Table 2 and k_2 can be determined by Eq. (12) of the same table. These equations showed that the decomposition reactions of xylose are lesser influenced by acid concentration that the sugar release reactions. The coefficient r^2 showed good values for all regressions.

Combining Eqs. (11) and (12) of Table 2 with the kinetic models, it is possible to predict the xylose concentration for any time and acid concentration over the range under consideration (0–300 min and 2–6% HCl). Fig. 2 shows how the generalized models predict the dependence of xylose concentration on HCl concentration and time. Several conditions achieved over 20 g xylose/l. Therefore, it is possible to select conditions leading to the highest xylose concentration and the minimum degradation product concentrations by comparing response surfaces for different products of sorghum straw hydrolysis. The generalized model predicted that maximum xylose concentration (21.3 g/l) can be achieved using 6% HCl after 83 min. This prediction compare very well with the reported for 122 °C (16.1 g xylose/l at 6% HCl and 70 min) (Herrera et al., 2003).

3.2. Kinetic modelling of glucose concentration

Eq. (5) was used to model the glucose concentration. $[G_0]$ cannot be obtained experimentally because the glucose released in the hydrolysis can proceed from both hemicellulosic and cellulose, therefore, $[G_0]$ has been introduced as a new regression parameter. Both values obtained by regression for $[G_0]$ and k_3 increased with the concentration (Table 1). The glucan susceptible of hydrolysis was in the range 4.0–5.1 g/l. This was lower than the range obtained with HCl at 122 °C (4.5–5.5 g/l) and with sulphuric acid at 80–122 °C (6.7–8.9 g/l). It can be inferred that the treatment with hydrochloric acid at 100 °C is more selective, being able to hydrolyse the hemi-

Fig. 2. Prediction of the generalized model for the dependence of xylose and glucose concentration on HCl concentration and time.

cellulosic fraction leaving cellulose and lignin in the solid phase. This is adequate for subsequent processing of the solid residue. The kinetic parameter k_3 increased with the HCl concentration. The values of r^2 showed a good agreement between experimental and predicted data and the values of F-test probability showed that the models are accurate. Fig. 1 shows the experimental and predicted data for these hydrolyses at each concentration of HCl.
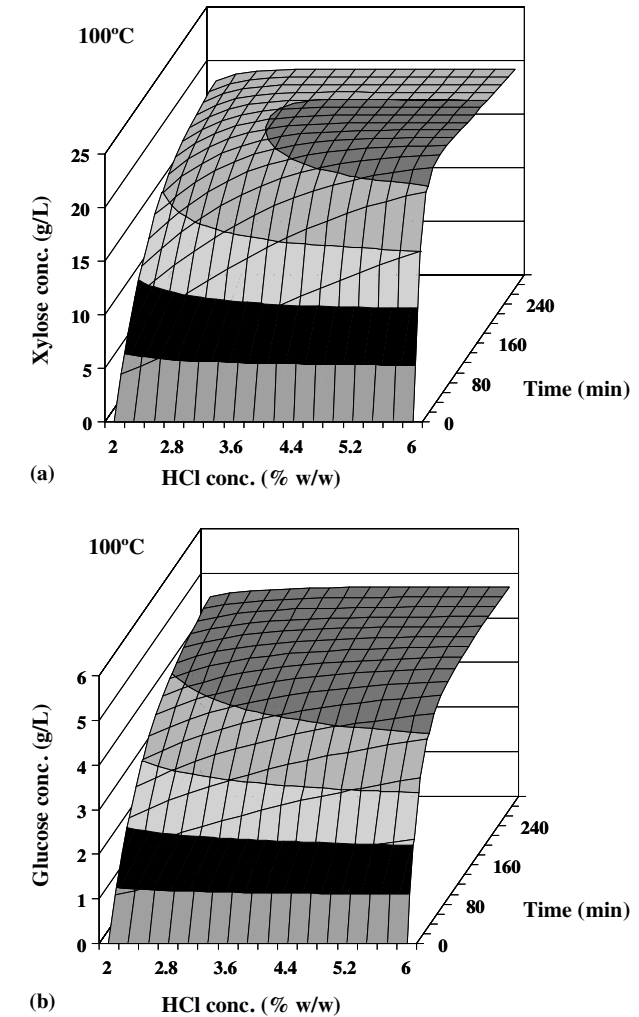
A generalized model for predicting glucose was also developed. Kinetic parameters k_3 were correlated with the HCl concentration (C_a) by mean of the empirical Eq. (13) showed in Table 2. Fig. 2 shows how the generalized models predict the dependence of glucose concentration on HCl concentration and time. It can be observed a slight dependence on HCl concentration. The models predict a maximum glucose concentration (4.7 g/l) at the most severe conditions (6% HCl and 300 min), but less than 4 g/l can be obtained after $t < 60$ min for any HCl concentration.

Table 2
Generalized models for the prediction of kinetic parameters of HCl hydrolysis of sorghum straw at 100 °C

Products	Models		r^2
Xylose generation	$k_1 = 0.00270 C_a^{1.70}$	(11)	0.9999
Xylose degradation	$k_2 = 0.0007 C_a^{-0.15}$	(12)	0.9476
Glucose	$k_3 = 0.0052 C_a^{0.97}$	(13)	0.9525
Furfural	$k_4 = 0.0443 C_a^{-0.64}$	(14)	0.9538
Acetic acid	$k_5 = 0.01476 C_a^{0.69}$	(15)	0.9044

3.3. Kinetic modelling of furfural concentration

Table 1 shows the kinetic and statistical parameters fitting the model for furfural generated in the hydrolysis of sorghum straw at 100 °C, whereas Fig. 1 shows the experimental and predicted data for furfural at different HCl concentrations. The coefficient k_4 and $[F_0]$ was the regression parameters. $[F_0]$ varied over the range 0.9–1.7 g/l. These values are lower than the values obtained using HCl at 122 °C (8.3–6.8 g/l) or using sulphuric acid (4 g/l). The values of r^2 and F-test probability showed a good fitting.

Eq. (14) of Table 2 was used for the generalized furfural modelling. Fig. 3 shows that the model predicts a maximum furfural concentration of 1.2 g/l at 2% HCl and 300 min. This predicting compare very well with the prediction for HCl at 122 °C (5.6 g furfural/l) and for sulphuric acid at 80–122 °C (3.4 g furfural/l). Since furfural is an inhibitor of the growth of microorganisms involved in the subsequent fermentation steps, condi-

tions to minimize the formation of furfural should be selected. The results showed that a treatment with HCl at 100 °C should be selected instead higher temperatures or sulphuric acid.

3.4. Kinetic modelling of acetic acid concentration

Table 1 also shows the kinetic and statistical parameters fitting the model for the acetic acid generated during the hydrolysis of sorghum straw at 100 °C and Fig. 1 shows the experimental and predicted data for acetic acid concentration at different HCl concentrations. $[Ac_0]$ varied over the narrow range 2.38–3.36 g acetic acid/l. In this case, the range was higher than using 122 °C (1.7–2.4 g acetic acid/l) or using sulphuric acid (1.5–1.5 g acetic acid/l). The values of the determination coefficients (r^2) and F-test probability confirm that the models fit very well. The values of k_5 showed an increase with HCl concentration. Eq. (15) of Table 2 was used for the generalized acetic acid modelling. Fig. 3 shows that the model predicts a stable concentration over 2.5 g acetic acid/l after $t > 80$ min for any HCl concentration. This values are higher than using HCl at 122 °C (1.9 g/l) or sulphuric acid (1.5 g/l).

3.5. Overall optimisation

If the hydrolysates of sorghum straw are going to be used as fermentation media and the solid residue have to be processed for glucose production, it is important to obtain sugar solutions (mainly xylose) with low concentration of inhibitors (furfural and acetic acid). The generalized models show that more than 20 g xylose/l can be obtained with reduced generation of furfural and a acetic acid concentration stable lower than 2.9 g/l. This acetic acid concentration is not enough to hinder the fermentation of the most microorganism, therefore the maximum xylose concentration (21.3 g/l) can be considered as optimum for the overall hydrolysis of sorghum straw with HCl at 100 °C. The operational conditions in the optimum were 6% HCl and 83 min. The models predict hydrolysates containing 21.3 g xylose/l, 4.7 g glucose/l, 0.8 g furfural/l and 2.8 g acetic acid/l.

3.6. Comparison of dilute acids

Several diluted acids has been applied to the hydrolysis of lignocellusic materials, being the sulphuric acid the most used. The efficiency of the HCl can be compared with it using the parameter efficiency (E). The efficiency as catalyst can be defined as the ratio:

$$E = \frac{X}{\sum I} \tag{16}$$

where X is the xylose concentrations in the hydrolysates (the main product wanted) and $\sum I$ is the sum of the
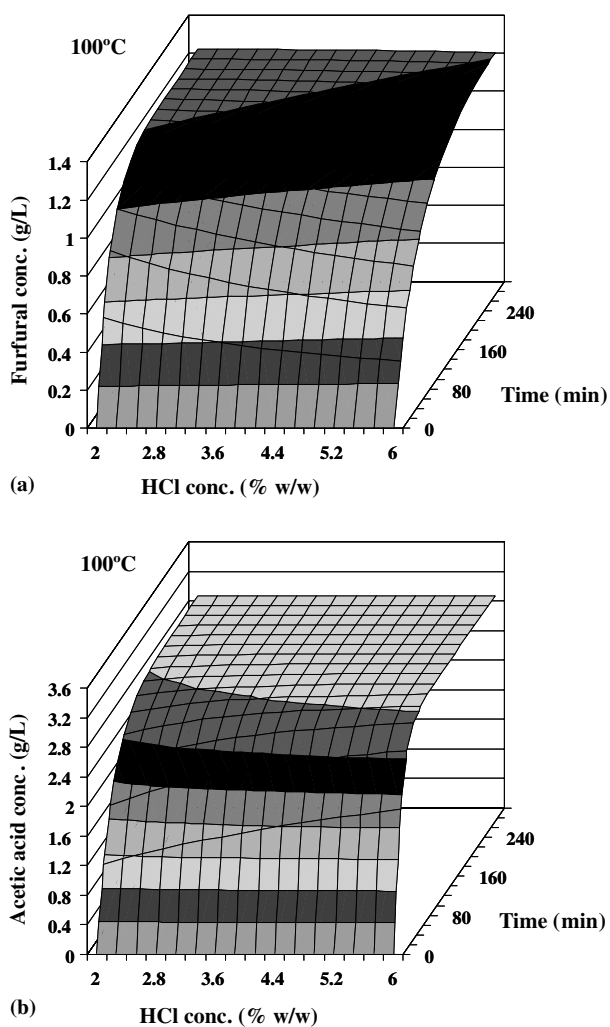
Fig. 3. Prediction of the generalized model for the dependence of furfural and acetic acid concentration on HCl concentration and time.

Table 3
Comparison of values of E (catalytic efficiency) for the selected optimum conditions in hydrolysis of sorghum straw and sugar cane bagasse

Raw material	Acid	Concentration (wt.%)	Temp. (°C)	Time (min)	E (g/g)	Reference
Sugar cane bagasse	H_2SO_4	2	122	24	3.02	Aguilar et al. (2002)
Sugar cane bagasse	HCl	2	128	51	2.54	Bustos et al. (2002)
Sorghum straw	H_2SO_4	2	122	71	1.98	Téllez-Luis et al. (2002b)
Sorghum straw	H_2SO_4	6	100	60	2.07	Téllez-Luis et al. (2002a)
Sorghum straw	HCl	6	122	70	2.10	Herrera et al. (2003)
Sorghum straw	HCl	6	100	83	2.67	This work

concentrations of all inhibitor in the hydrolysates (acetic acid and furfural) and the byproducts (glucose). Table 3 shows different values of E obtained from literature and the value obtained in the optimum for HCl at 100 °C. For comparative purpose, values of E obtained in the hydrolysis of sugar cane bagasse were also included.

Therefore, on the basis of values of E, a direct comparison can be done to determine the effect of the kind of acid in the hydrolysis.

Table 3 shows that HCl is more efficient as catalyst in the hydrolysis of sorghum straw than sulphuric acid. The efficiency at 100 °C was also higher than at 122 °C for both acids. However, Table 3 also shows that the efficiency depend on raw material. For instance, HCl is lesser efficient in the hydrolysis of sugar cane bagasse than sulphuric acid. It can be concluded that the acid more efficient and selectivity for the hydrolysis of sorghum straw is HCl at 100 °C.

Acknowledgements

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