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Fractionation of *Cynara cardunculus* (Cardoon) Biomass by Dilute-Acid Pretreatment

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Abstract

Cynara cardunculus L. (cardoon) is a Mediterranean perennial herb offering good potential as substrate for sustainable production of bioethanol. In this work the first approach to the study of dilute-acid pretreatment of cardoon biomass for biological conversion was made. The influence of temperature (160–200°C), acid concentration (0–0.2% [w/w]), and solid concentration (5–10% [w/v]) in the formation of free sugars and sugar decomposition products in the prehydrolyzate was studied using a response surface methodology. Results show a negative interaction effect between acid concentration and temperature in xylose recovery yield in prehydrolyzate, whereas dry matter concentration does not exert a significant effect. Xylose recovery yield reaches a maximum of about 80% of the content in dry untreated raw material at 180°C and 0.1 or 0.2% acid addition. At these conditions the ratio of monomers found in prehydrolyzate in relation to total sugar yield for xylose is close to 100%. Furfural concentration, the major furan determined in the prehydrolyzate, increases as pretreatment severity rises. Maximum furfural yield of 4.2 g/100 g dry untreated raw material was found at 200°C and 0.2% acid concentration. The yield of furfural at the conditions in which maximum xylose recovery is attained is substantially lower, less than 2 g/100 g dry untreated raw material. This fact supports the idea of using moderate temperatures in dilute-acid processes, which at the same time provides reasonably high sugar recovery yield and avoids high inhibitory products formation.

Index Entries: Acid prehydrolyzate; furfural; pretreatment optimization; surface-response methodology; xylose recovery; herbaceous crop.

Introduction

Cardoon (*Cynara cardunculus* L.) is a dicotyledonous herb originally from the Mediterranean area that can be considered as a potential lignocellulosic feedstock for biofuels production in Spain owing to its good adaptability to the environmental conditions of the country—hot and dry

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climates—and high biomass productivities *(1)*. This crop is traditionally cultivated in Spain for food purposes in a shorter artificial cultivation cycle to produce edible blanched leaf petioles using intensive crop management practices. However, it can be cultivated for biomass production in accordance with its natural autumn–spring cycle and whole aboveground biomass can be harvested annually for several years.

Cardoon biomass presents a wide range of applications as raw material for the production of fuels and high-value products. The whole plant has been tested as solid biofuel for power and heat generation *(2)*, seeds for oil production, *(3)* and stalks as substrate for paper pulping *(4)*. Another alternative use for the aerial part of the plant (stalks and branches) would be as substrate for sustainable production of transportation fuels and chemicals that are now primarily made from petroleum. Some preliminary studies performed by Martinez et al. *(5)* on cardoon biomass showed good prospects as substrate for biological conversion into ethanol or other products, but more information is needed to establish its real potential.

Regarding biological processing of cellulosic biomass, it is well-known that each type of feedstock, whether wood or agricultural residue, requires a particular pretreatment to minimize the degradation of the substrate and maximize the overall sugar yield. When enzymatic hydrolysis is involved in the process, a pretreatment step is vital to effectively prepare cellulose for enzymes action and provide high sugar yields. So, pretreatment has been frequently highlighted as one of the most costly process steps having a major influence on both previous (e.g., size reduction) and subsequent operations (e.g., enzymatic hydrolysis and fermentation) *(6)*.

There are numerous pretreatment methods or combinations of pretreatment methods available to fractionate lignocellulosic biomass. Among the chemical pretreatment processes used for cellulosic feedstock (dilute acid, alkaline, organic solvent, ammonia, sulfur dioxide, or carbon dioxide), considerable research effort has been carried out on acid catalyzed hydrolysis to depolymerize the hemicellulose fraction contained in biomass *(7,8)*. Xylose, glucose, and other sugars are released in the liquid stream while providing a cellulose-enriched solid more susceptible to further enzymatic hydrolysis. Dilute acid at moderate temperatures has been demonstrated to effectively remove and recover most of the hemicelluloses as dissolved sugars in lignocellulosic substrates. Dilute-acid pretreatment has the advantage of not only solubilizing hemicellulose, but also converting solubilized hemicellulose to fermentable sugars *(9)*. However, with sustained hydrolysis the sugars may be degraded to decomposition products such as furfural and hydroxymethylfurfural (HMF) that lower the sugar yield and affect the subsequent fermentation step. So, to produce fermentable prehydrolyzates and to prevent high losses in yields, it is necessary to choose process conditions that keep at a low level the amount of degradation products produced.

This work represents a first approach to the assessment of dilute-acid pretreatment for biological conversion of *C. cardunculus* biomass into ethanol. In this article, the influence of pretreatment parameters (temperature, acid concentration, and solid concentration) in the formation of free sugars and sugar decomposition products in the prehydrolyzate was investigated by using a statistical experimental design. This statistical model involves fitting an empirical model to the experimental data and identifying the optimal temperature, acid concentration, and solid concentration in the pretreatment stage by using response surface technique.

Materials and Methods

Raw Material

Cardoon biomass (stalks and branches) (6% moisture content) was obtained from Agroenergy Group of the High School of Agricultural Engineering of Madrid (Spain). Biomass was milled to a particle size smaller than 5 mm using a laboratory hammer mill (Retsch GmbH & Co. KG, Germany), homogenized and stored until used. As the presence of high amounts of alkaline ash in herbaceous feedstock has been reported that can partially neutralize the sulfuric acid and lower the acidity of the reaction mixture in acid pretreatments, the buffering capacity of cardoon biomass in the aqueous phase was determined. The pH of 1% (w/w) sulfuric acid solution was measured before and after mixing with the biomass in the same ratio of substrate, acid, and water as in pretreatment runs. Neutralizing capacity was calculated based on the change of pH of acid solution according to Esteghalian et al. *(10)*.

Pretreatment

Cardoon biomass samples were pretreated in a 2-L stainless steel Hastelloy-C stirred reactor (Model EZE-Seal, Autoclave Engineers, Erie, PA). Process parameters tested were temperature (160–200°C), solid concentration in the reactor (5–10% [w/v]), and acid concentration in the reaction mixture (0–0.2% [w/w]). Biomass samples were loaded into the reactor at the different solid/liquid ratios and heated at selected temperatures by an external heating jacket. The heating rate was between 2 and 4°C/min. When desired temperature was reached, the corresponding amount of sulfuric acid was added to provide final acid content in the reactor, taking into account the buffering capacity of biomass previously determined. After 10 min pretreatment time, the reactor was removed from the heating jacket, and cooled to about 40°C in less than 10 min. The wet material was vacuum-filtered and separated into a water insoluble fraction (WIS) and a filtrate or prehydrolyzate fraction, which contains sugars, furfural, HMF, and other degradation products. Both fractions were analyzed to determine their chemical composition as described under "Analytical Procedures".

Solid recovery yield was then calculated as dry weight of WIS remaining after pretreatment referred to 100 g of dry untreated raw material.

Analytical Procedures

Raw Material and Pretreated Substrates Composition

The chemical composition of raw material and WIS fraction from pretreatment was determined using the standard laboratory analytical procedures for biomass analysis described by the National Renewable Energy Laboratory (Colorado) *(11)*.

Prehydrolyzate Composition

The sugar content of prehydrolyzate after pretreatment was determined "as is" and later by performing a mild acid hydrolysis (3%[v/v] H_2SO_4, 120°C, 30 min), measuring glucose, xylose, arabinose, galactose, and mannose concentration by high-performance liquid chromatography (HPLC) in a Waters 2695 liquid chromatograph (Waters, Milford, MA) with refractive index detector. A CARBOSep CHO-682 LEAD column (Transgenomic, Omaha, NE) operating at 80°C with Milli-Q water (Millipore, Billerica, MA) as mobile-phase (0.5 mL/min) was used.

Furfural and HMF, vanillin, syringaldehyde, catechol, cumaric acid, and ferulic acid analyses were performed by HPLC (Hewlett Packard, Palo Alto, CA), using an Aminex ion exclusion HPX-87H cation exchange column (Bio-Rad, Hercules, CA) at 65°C. Mobile phase was 89% H_2SO_4 5 m*M* and 11% acetonitrile at a flow rate of 0.7 mL/min. Column eluent was detected with a 1040A photodiode-array detector (Agilent, Waldbronn, Germany). Acetic, formic, and levulinic acid were quantified by HPLC with a 2414 Waters refractive index detector. A Bio-Rad Aminex HPX-87H (Bio-Rad, Hercules, CA) column maintained at 65°C with a flow rate of 0.6 mL/min was used. Mobile phase was H_2SO_4 (5 m*M*).

Statistical Experiment Design

A response surface methodology was used to study the effects of temperature, acid concentration, and solid concentration in the formation of free sugars and degradation products in the prehydrolyzate. A Box-Behnken design with four center points was created and evaluated with commercial software Statgraphics 5.0 (Manugistics Inc., Rockville, MD). This design allows estimation of the main effects and two factor interactions using analysis of variance. The response surface is calculated using a quadratic polynomic model. The parameters were tested at two levels: temperature (160 and 200°C), acid concentration (0 and 0.2% [w/w]), and solid concentration (5 and 10% [w/v]). Reaction time was fixed at 10 min. The experimental error was estimated in the center point (180°C, 0.1% acid and 7.5% solid content), which was carried out four times. The conditions for each experiment, which were performed in fully randomized order, are shown in Table 1.

Table 1
Conditions for Dilute-Acid Pretreatment of Cardoon Biomass According to Experimental Design

Experiment	Code	Temperature (°C)	Solid/liquid ratio (w/v [%])	Acid concentration (w/w [%])
1	(–1 –1 0)	160	5	0.1
2	(1 –1 0)	200	5	0.1
3	(–1 1 0)	160	10	0.1
4	(1 1 0)	200	10	0.1
5	(–1 0 –1)	160	7.5	0
6	(1 0 –1)	200	7.5	0
7	(–1 0 1)	160	7.5	0.2
8	(1 0 1)	200	7.5	0.2
9	(0 –1 –1)	180	5	0
10	(0 1 –1)	180	10	0
11	(0 –1 1)	180	5	0.2
12	(0 1 1)	180	10	0.2
13	(0 0 0)	180	7.5	0.1
14	(0 0 0)	180	7.5	0.1
15	(0 0 0)	180	7.5	0.1
16	(0 0 0)	180	7.5	0.1

Results and Discussion

Raw Material Composition

The chemical analysis of raw material showed the following composition (dry weight [%]): cellulose, 33.8; hemicellulose, 18.5 (xylans 14.7, arabinans 1.2, galactans 2.0, and mannans, 0.6); acid insoluble lignin, 14.0; acid soluble lignin, 2.4; acetyl groups, 3.8; ash, 6.6; and extractives, 14.3 (total 93.4%). The high ash content of 6.6% suggested a buffering capacity of cardoon biomass in the aqueous phase of dilute-acid pretreatment. The neutralizing ability of cardoon biomass in aqueous phase was determined as 36.0 mg H_2SO_4/g dry substrate and it was used to correct the amount of sulfuric acid added in pretreatment experiments. This value is consistent with the high values found in other herbaceous species such as those reported by Esteghalian et al. *(9)* for switch grass and corn stover biomass: 43.7 and 25.8 mg H_2SO_4/g dry substrate, respectively. Lower buffering capacities less than 10 mg H_2SO_4/g dry substrate have been reported for hardwoods *(12)*, in agreement with the lower ash content of this type of biomass.

Sugar Yield in Prehydrolyzate

The monosaccharide yields in prehydrolyzate from dilute-acid pretreatment experiments of cardoon biomass are shown in Table 2. Values correspond to sugar yield (gram/100 g dry untreated raw material) after mild acid posthydrolysis of prehydrolyzate, so include both oligomers and

Table 2
Sugar Yield in Prehydrolyzate, Solid Fraction (WIS) Composition, and pH and Solid Recovery Values From Dilute-Acid Pretreatment of Cardoon Biomass

Acid concentration (w/w [%])	0				0.1					0.2			
Temperature (°C)	160	180		200	160		180[a]	200		160	180		200
Solid concentration (w/v [%])	7.5	5	10	7.5	5	10	7.5[a]	5	10	7.5	5	10	7.5
Prehydrolyzate sugar yield (gram/100 g raw material)													
Glucose	0.4	0.4	0.4	0.4	0.5	0.6	1.4	5.1	3.8	1.0	2.8	2.8	7.0
Monomeric form (%)	34	47	76	27	96	85	98	84	90	100	94	100	98
Xylose	0.9	8.0	8.9	3.7	4.7	5.9	13.5	10.4	5.7	11.1	13.5	12.4	6.3
Monomeric form (%)	0	2	2	37	83	88	97	84	94	92	96	100	100
Galactose	0.9	1.3	1.3	0.5	1.2	1.4	2.1	1.7	0.8	1.9	2.3	2.1	1.3
Arabinose	0.6	0.6	0.4	0.04	1.3	1.2	1.4	0.8	0.2	1.6	1.1	1.0	0.8
Mannose	0.05	0.04	0.08	0.25	0.2	0.1	0.5	0.7	0.3	0.3	0.6	0.5	0.5
Total sugars	2.8	12.3	13.1	4.9	7.9	9.2	18.9	18.7	10.8	15.9	20.3	18.8	15.9
pH	4.36	4.19	4.08	3.79	1.86	1.81	1.80	1.81	1.92	1.56	1.36	1.52	1.66
WIS composition (w/w [%])													
Glucan	42.7	53.0	56.1	58.5	55.8	56.1	64.1	62.5	60.7	60.2	65.3	63.4	62.8
Xylan	16.2	9.4	7.5	5.8	14.6	13.3	3.3	0	0	5.6	1.4	1.1	0
Lignin	19.4	19.8	21.1	27.9	21.4	23.0	26.0	27.5	29.7	23.5	25.4	27.4	31.2
Solid recovery (g/100 g dry untreated raw material)	82.7	64.7	64.2	58.6	61.9	63.5	54.6	47.4	50.2	57.8	49.8	51.1	44.1

[a]Average values of four center points.

monomers. Soluble sugar monomers ratio, calculated from sugar measurement before such hydrolysis, is shown for glucose and xylose, which are the major sugars present in prehydrolyzate.

Results show that, although significant amounts of xylose can be recovered in prehydrolyzate following dilute-acid pretreatment, xylose recovery is highly dependent on pretreatment conditions. Xylose is the major sugar found in prehydrolyzate, accounting for up to 65–70% of total sugars. Arabinose, glucose, galactose, and mannose are present at lower concentration in most experiments, especially at low temperature and acid concentration. Xylose recovery yield in relation to the content in dry untreated raw material (16.6 g/100g) reached a maximum of about 80% at 180°C and 0.1 or 0.2% acid addition, regardless of solid concentration tested. Higher and lower temperatures led to decreased recovery yields values, depending on acid concentration. Glucose was found in significant amounts at the highest temperature of 200°C in experiments performed with acid, reaching recovery yields from 10% to 20% of initial content (37.2 g/100 g dry untreated raw material). This fact suggests slight cellulose hydrolysis at increased severities.

On the other hand, the ratio of monomers found in prehydrolyzate in relation to total sugar yield for xylose is quite high in all experiments performed with acid (83–100%). The lower pH found in these experiments (Table 2) facilitates the complete depolymerization of oligomeric hemicelluloses. Glucose oligomers were highly hydrolyzed in the presence of diluted acid (95–100% in monomeric form), although considerable amounts of monomers (30–75%) were also found even in nonacid experiments at 180°C. Although for mass balance purposes it is assumed that all glucose is derived from cellulose depolymerization, this easily hydrolyzed glucose could come from depolymerization of xyloglucans, which have been described to make up dicotyledons hemicelluloses *(13)*.

The significance of the effect of temperature, acid concentration, and solid concentration on xylose recovery was determined by analysis of variance and the results can be visualized in standardized Pareto chart shown in Fig. 1A. A negative interaction effect between acid concentration and temperature occurs, which causes different responses at increasing acid concentration depending on temperature tested. Dry matter concentration does not exert a significant effect in xylose recovery, within the limits of the experimental design. Figure 1B shows response surface for xylose recovery in prehydrolyzate at a fixed solid concentration of 7.5% (w/v), calculated using a quadratic polynomic equation. From this graph it can be deduced that conditions resulting in maximum xylose recovery are 180°C and acid concentration close to 0.2%. It also illustrates the aforementioned interaction between acid concentration and temperature, so that the positive effects of increasing acid at lower temperature of 160°C (from 4.7% at 0.1% acid to 11.1% at 0.2% acid) cannot be observed at 200°C, wherein yields even decrease (from 10.4% at 0.1% acid to 6.3% at 0.2% acid).

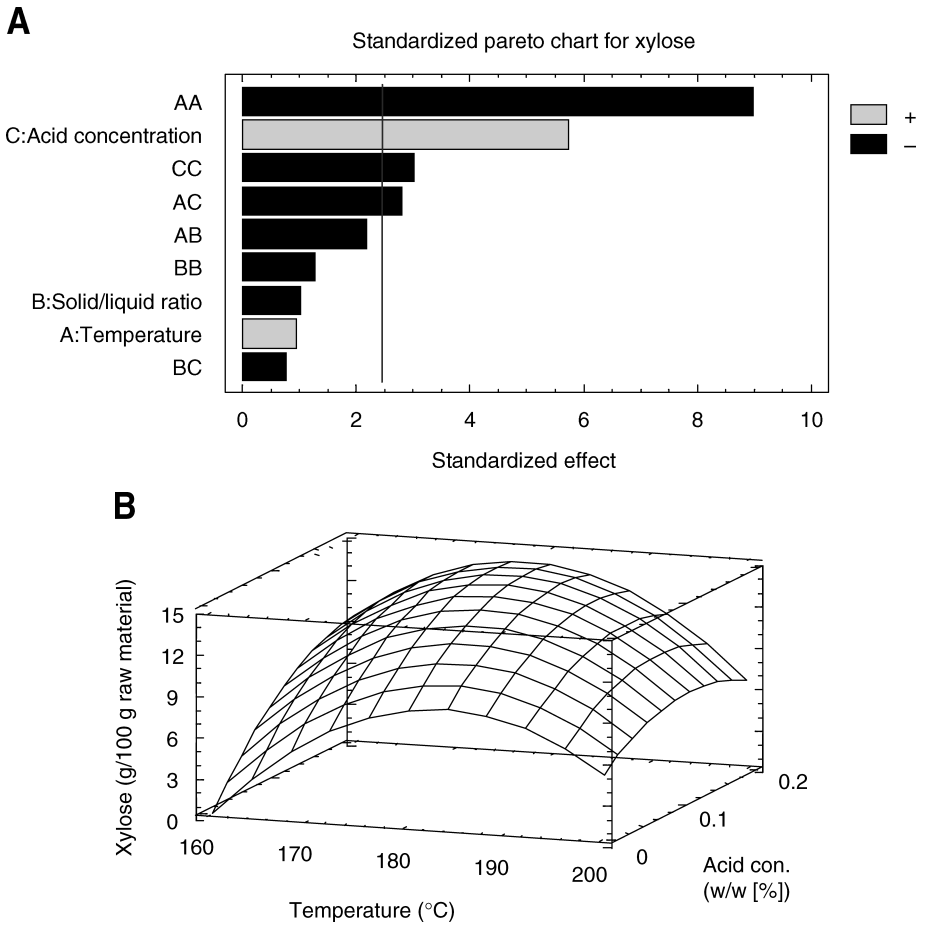

Fig. 1. Standardized Pareto chart **(A)** and estimated response surface **(B)** for xylose recovery yield. Solid concentration is fixed at central value (7.5%, [w/v]).

The effectiveness of dilute-acid hydrolysis in xylan depolymerization has been reported in other lignocellulosic herbaceous substrates. Although it is difficult to compare results among different raw materials, xylose yields obtained in this study are similar to those reported in dilute-acid hydrolysis of rice straw (2–15 g/100 g raw material) *(14)* or sugarcane bagasse (9–14 g/100 g raw material) *(15)* in experiments performed in batch hydrolysis reactors. Esteghalian et al. *(9)* reported 80% xylose recovery at temperatures between 170–190°C and 1% (w/w) acid concentration when pretreating corn stover in a Parr reactor similar to that used in this study. Our data of high monomer ratio found in prehydrolyzates obtained in acidic conditions support the idea that satisfactory hydrolysis of cardoon hemicelluloses to monosaccharides can be achieved in a single step reaction within the limits of the selected experimental design, although it is essential to consider degradation products formation.

Degradation Products in Prehydrolyzate

Furfural and HMF are well-known byproducts formed in acid-hydrolysis of lignocellulosic materials originating from pentoses and hexoses degradation in acidic conditions. These compounds can be further degraded to formic and levulinic acid or they can polymerize *(15)*. Moreover, during pretreatment acetic acid is released from hydrolysis of acetyl groups present in hemicelluloses, as a consequence of deacetylation of acetylated pentosans. The amount of these compounds found in prehydrolyzates varies greatly depending on the nature of lignocellulosic substrate and the process conditions. Figure 2 shows the yield of sugar degradation compounds and aliphatic acids in prehydrolyzates from dilute-acid hydrolysis of cardoon biomass at different process conditions. Furfural is the most important furan found in prehydrolyzate (Fig. 2A), which is consistent with the major presence of xylan in hemicelluloses of cardoon biomass (approx 90%). Results show that the yields of both furan compounds increase as the severity of pretreatment rises, attaining maximum values of 4.2 and 0.7 g/100 g dry untreated raw material for furfural and HMF, respectively, at 200°C and 0.2% acid concentration. The yield of furfural at conditions where maximum xylose recovery is attained (180°C and acid concentration close to 0.2%) is substantially lower, less than 2 g/100 g dry untreated raw material.

From statistical analysis of the effect of process parameters on furfural yield, it can be deduced that there is a significant positive interaction between temperature and acid concentration (Fig. 3A). The effect of increasing acid concentration results in considerably higher furfural yield at 200°C; conversely furfural is hardly detectable at 160°C. This fact supports the idea of using moderate temperatures in dilute-acid processes, which, at the same time as providing reasonably high sugar recovery yield, also avoids excessive inhibitory products formation. A complete prevention of sugar degradation products can be achieved by lowering pretreatment temperature below 150°C, but longer reaction times are needed and enzymatic saccharification of pretreated substrate suffers from poor results. Saha et al. *(8)* reported good hemicelluloses solubilization yields without further degradation in dilute-acid pretreatment of rice hulls at 1% (v/v) H_2SO_4 and 121°C for 1 h, although the enzymatic saccharification yield of the pretreated slurry remained at 60% based on total carbohydrate content.

The formation of aliphatic acids (acetic, formic, and levulinic) in prehydrolyzate after dilute-acid pretreatment of cardoon biomass is shown in Fig. 2B. The yield of acetic acid fluctuates from low values less than 1 g to a maximum close to 5 g/100 g dry untreated raw material, depending on the severity of pretreatment. As shown in the analysis of raw material, an elevated proportion of acetyl groups are present in hemicelluloses of cardoon biomass and consequently, acetic acid is the prevailing acid present in prehydrolyzate. The maximum amount of acetic acid corresponded to 90%

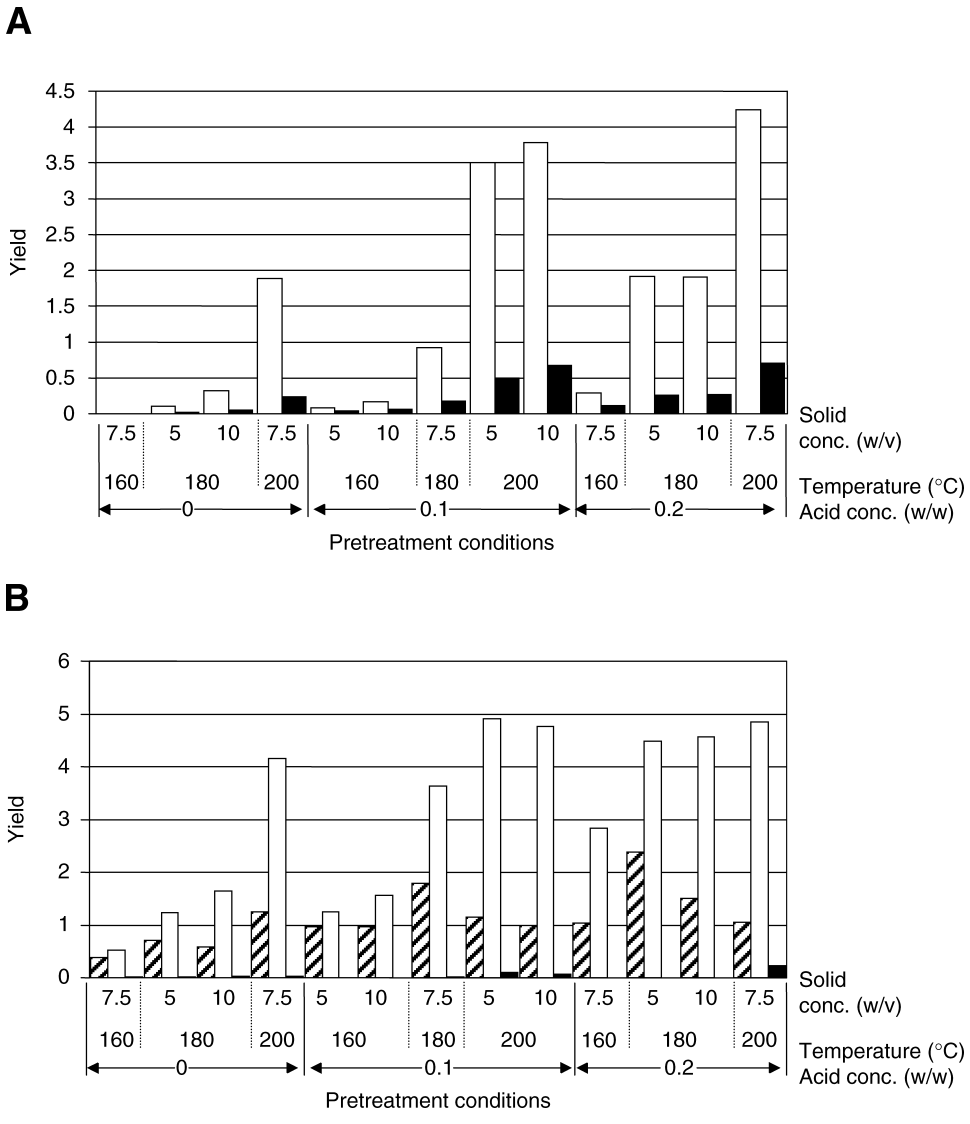

Fig. 2. Yields of furfural (□) and HMF (■) **(A)** and formic (▨), acetic (□), and levulinic acid (■) **(B)** in prehydrolyzates from dilute-acid pretreatment of cardoon at different conditions. Results are reported as gram/100 g dry untreated raw material.

of the acetyl groups determined in raw material, which indicates almost complete hydrolysis under the conditions tested. Regarding the effect of process conditions in acetic acid recovery, Fig. 3B illustrates the response surface graph showing a positive effect of the temperature and acid concentration on the formation of acetic acid. The effect of temperature is stronger as even in nonacid conditions increasing acetic acid is formed as temperature rises from 160 to 200°C.

Formic acid, yielding up to 2.5 g/100 g dry untreated raw material, is detected in all experiments, whereas levulinic acid is almost negligible

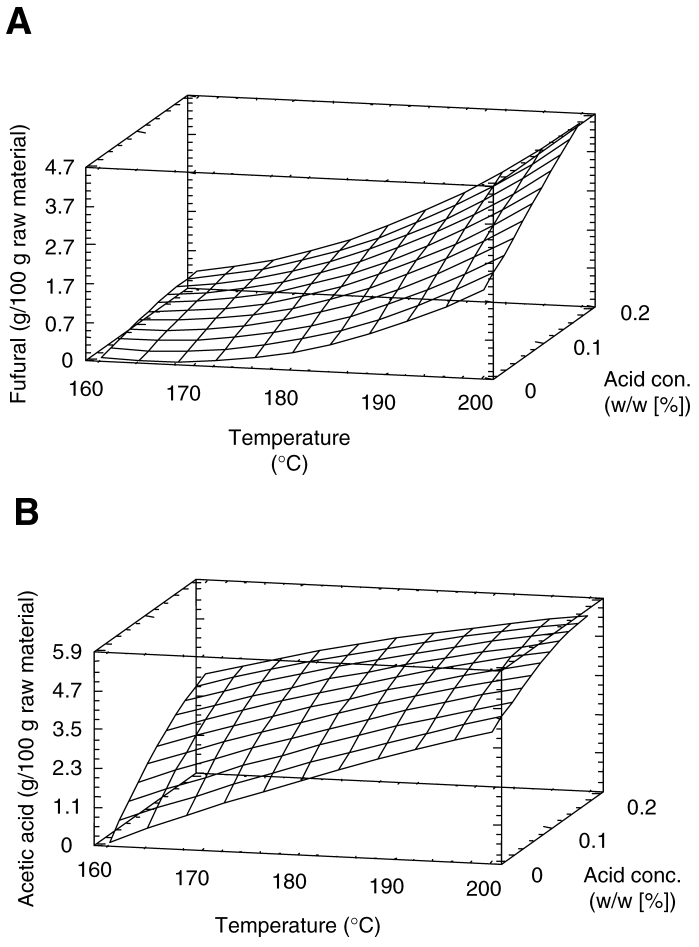

Fig. 3. Estimated response surface for furfural **(A)** and acetic acid **(B)** yields. Solid concentration is fixed at central value (7.5%, [w/v]).

(Fig. 2B). As HMF is broken down to equimolar amounts of levulinic and formic acids and HMF yield is rather low (Fig. 2A), most of the formic acid formed probably comes from furfural degradation. The content of some aromatic monomers coming from lignin degradation was also determined. Results of catechol, vanillin, and syringaldehyde in the different experiments are shown in Table 3. The most severe conditions of temperature and acid led to increased yields of catechol, the major phenolic monomer measured in all conditions tested. A higher ratio of syringil derivative (syringaldehyde) was found in relation to guayacil (vanillin). At low severity conditions of 160–180°C without acid, these compounds were not found. Aromatic acids were not detected in any prehydrolyzate.

The maximum yield of soluble lignin-derived compounds, 196 mg/100 g dry untreated raw material, was obtained at 200°C, 5% solid concentration and 0.1% acid concentration. Total concentration in acid prehydrolyzates

Table 3
Yield of Phenolic Compounds (mg/100 g Dry Untreated Raw Material) in Prehydrolyzate From Dilute-Acid Pretreatment of Cardoon Biomass

Acid concentration (% w/w)	0			0.1			0.2						
Temperature (°C)	160	180		200	160	180	200	160	180		200		
Solid concentration (% w/v)	7.5	5	10	7.5	5	10	7.5	5	10	7.5	5	10	7.5
Catechol	2.6	8	10	39	12	20	61	96	80	39.9	86	83	85.3
Vanillin	nd	4	nd	13	8	6	15	20	10	4	nd	20	26.6
Syringaldehyde	nd	nd	nd	13.3	17.2	14	36	80	70	nd	nd	60	60.6

nd, detected.

ranged from low values about 40 mg/L at 160°C to 196 mg/L at 200°C. Concentrations found in prehydrolyzates produced at 180°C (about 80 mg/L) are lower than those reported by Fenske et al. *(16)* on the aromatic composition of dilute-acid prehydrolyzates prepared from switch grass, corn stover, and poplar biomass at 180°C, 10 min residence time and 1% w/w acid concentration (112.4, 140.8, and 247.3 mg/L). Our results support the idea of the lower toxicity of prehydrolyzates from herbaceous feedstocks in comparison with wood-derived ones, which implies a clear advantage from the point of view of its fermentation.

Mass Balance on Xylan

To complete the assessment of the effectiveness of dilute-acid pretreatment to fractionate cardoon biomass, an overall mass balance for xylose, the major hemicellulose-derived sugar present in cardoon biomass, was carried out (Fig. 4). For this purpose, xylose measured in different fractions from pretreatment (WIS and prehydrolyzate) and furfural content were considered. Xylose recovery yields in WIS and prehydrolyzate (gram xylose/gram xylose in dry untreated raw material) were calculated based on data presented in Table 2. For purposes of mass balance, all furfural found in prehydrolyzate (Fig. 2) was considered to come from xylose degradation. Xylose recovery yield as furfural (gram furfural × 1.56/g xylose in dry untreated raw material) was calculated and summed up to WIS and prehydrolyzate xylose recovery values, as illustrated in Fig. 4.

Results show that at 180°C and 0.1 or 0.2% acid addition, regardless of the solid concentration, a 100% mass closure is attained. At these conditions, the percent of solubilized xylose amounts to 82%. At lower temperatures of 160 and 180°C in experiments without acid addition and at 160°C with 0.1% acid, the mass closure reaches values close to 90%, although a high proportion of xylan remains in the WIS fraction. Contrarily, the highest temperature of 200°C leads to overall recovery decreasing from 78% to 60%

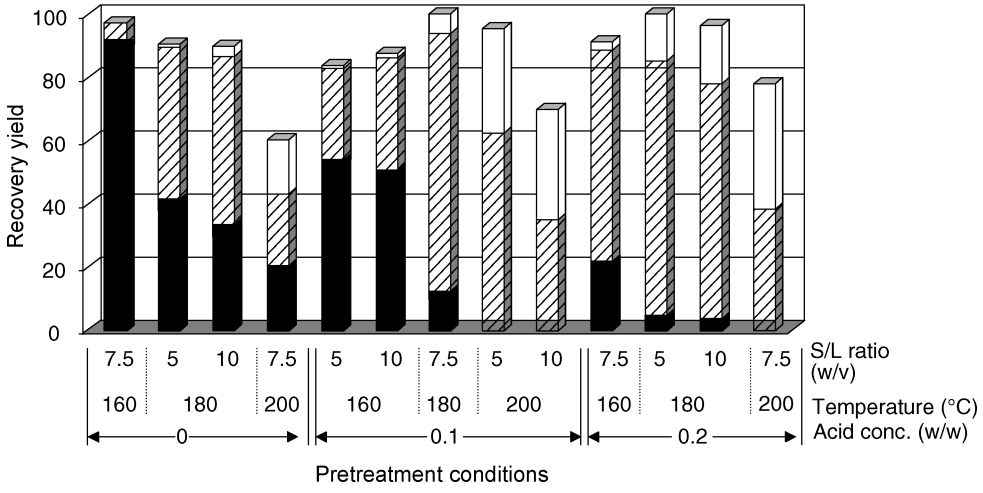

Fig. 4. Xylose recovery yields in WIS fraction (■), prehydrolyzate (▨), and xylose recovery as furfural (□), in prehydrolyzates from dilute-acid pretreatment of cardoon at different conditions. Results are reported as gram xylose/100 g xylose in dry untreated raw material.

with increased furfural formation and xylose losses, even in experiments without acid. Summing up, from results obtained in this work it can be concluded that mild temperature of 180°C with the addition of 0.1% (w/w) would be the condition chosen to effectively remove and recover most of the hemicelluloses as dissolved sugars in cardoon biomass. Nevertheless, the presence of inhibitory compounds makes necessary further studies on the fermentability of prehydrolyzate to determine the possibilities of using this fraction, or the whole slurry, for fermentation.

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