Industrial Crops and Products 40 (2012) 39–44

Contents lists available at SciVerse ScienceDirect

Industrial Crops and Products

journal homepage: www.elsevier.com/locate/indcrop

Evaluation of antioxidant activity of dilute acid hydrolysate of wheat straw during xylose production

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ARTICLE INFO

Article history:
Received 12 November 2011
Received in revised form 1 February 2012
Accepted 24 February 2012

Keywords:
Xylose
Xylitol
Antioxidant
Phenolic

ABSTRACT

Wheat straw, a lignocellulosic waste material, can be used as a raw material for the production of high-value products such as xylose for xylitol production or phenolic compounds that have antioxidant activity. There is a growing interest in the use of lignocellulosic wastes for conversion into various chemicals because of their low cost and the fact that they are renewable and abundant. The objective of this study was to determine the effects of H_2SO_4 concentration, temperature and reaction time on the production of sugars (xylose) and phenolic compounds. Response surface methodology (RSM) was used to optimize the hydrolysis process to obtain high xylose yield and phenolic compounds. The optimum reaction temperature, reaction time and acid concentration were 120 °C, 45 min and 4.7% respectively. Under these conditions, xylose and phenolic of the hydrolysate was found to be 0.16 g/g-wheat straw and 0.014 g-gallic acid/g-wheat straw, respectively.

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1. Introduction

Mild acid treatments of lignocellulosic materials in the presence of mineral acid (which acts as a catalyst) are currently used for converting the hemicellulosic fraction of lignocellulose into monosaccharides. The main component of the hemicellulosic fraction is xylan, a heteropolysaccharide with homopolymeric backbone of xylose units, which can be a source for production of chemicals, including food-related products, such as xylose or xylitol (Saha, 2003). Acid hydrolysis process not only breaks down the hemicellulose to monosaccharides, but also cleaves the β-1-4 alkyl-aryl linkages in lignin and lignin-hemicellulose linkages to form soluble phenolic compounds (Garrote et al., 2004; Nabarlatz et al., 2007). Lignin is either covalently linked to polysaccharides via sugar residues, or phenolic acids esterify to polysaccharides. Although most of the lignin is acid-insoluble (klason lignin), a part of it can be solubilized in acidic media (acid-soluble lignin). While hot water can extract the free phenolic acids, acid hydrolysis can release simple esterified phenolic acids. These phenolics, considered as the byproducts of acid hydrolysis of lignocellulosic materials, have potential application as food additives with antioxidant activity (Moure et al., 2008). The acid-soluble lignin fractions such as *p*-hydroxybenzoic acid, ferulic acid, vanillic acid, syringic acid, coumaric acid, syringaldehyde, *p*-hydroxybenzaldehyde and vanillin are well known. Among them, ferulic acid has received enormous attention due to its potential application in food (preservative agent, gel-forming properties, flavor precursor), health (antioxidant, antimicrobial, anti-inflammatory) and cosmetic (photo protecting agent) industries (Barberousse et al., 2009).

Hemicellulose hydrolysis of different lignocellulosic materials using dilute acid has been studied by many researchers (Rahman et al., 2007; Roberto et al., 2003; Canettieri et al., 2007). The results showed that the yield of sugar and sugar dehydration products such as furfural and soluble phenolic compounds is dependent on the type of material and the operational conditions (measured by the severity factor) (Rahman et al., 2007). The presence of phenolic compounds and sugar degradation products with xylose is undesirable because they either decrease the purity of xylose or inhibit its microbial metabolism. To overcome this problem, it is necessary to run the hydrolysis reaction at less severe conditions to keep the degradation products at low concentration.

Wheat straw is one of the most widely distributed and abundant lignocellulosic waste found in Turkey (Bascetincelik et al., 2006). Utilization of wheat straw for industrial purposes as a renewable material for the production of value-added products is receiving interest due to its huge amount of polymers (cellulose and hemicellulose), low cost, wide availability and reduction of environmental pollution.

The aim of the present investigation was to evaluate the effect of operational conditions (temperature, time and acid concentration)

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doi:10.1016/j.indcrop.2012.02.035

on the production of sugars (xylose, glucose, arabinose and sugar dehydration products) and phenolic compounds, and determine the antioxidant activity of acid hydrolysate of wheat straw.

2. Materials and methods

2.1. Materials

Wheat straw was collected from local farmers in Turkey, air-dried and milled to obtain particles of 1–5 mm length and 1 mm thickness. Aminex HPX 87H column (dimension: 300 mm × 7.8 mm; average particle size: 9 μm) and cation H cartridge were purchased from Bio-Rad Laboratories, CA, USA. All the chemicals were of analytical grade obtained either from Sigma Chemical Company, MO, USA or Merck KGaA, Germany.

2.2. Acid hydrolysis

Hydrolysis experiments were performed in a 100-ml stainless-steel pressure batch reactor. The reactor was loaded with 2 g of wheat straw and 20 ml of sulfuric acid solution. The reactions were carried out in the range of 100–140 °C under different sulfuric acid concentrations (2–6% H_2SO_4) and residence times (15–45 min). After the reaction was complete, the solid material was separated by filtration and the filtrate was analyzed for xylose, glucose, acetic acid and furfural.

Hydrolysate samples were analyzed using HPLC system equipped with a refractive index detector (Perkin Elmer Series 200), and column oven (Perkin Elmer Series 200) on Aminex HPX 87H (300 mm × 7.8 mm), which was preceded by its complimentary cation H cartridge on Aminex HPX 87H (300 mm × 7.8 mm), which was preceded by its complimentary cation H cartridge. Sugars and acetic acid were eluted with 5 mmol/l H_2SO_4 from the column at 45 °C with a flow rate of 0.5 ml/min (Canettieri et al., 2007). A complete analysis was carried out in 70 min. Their concentration was quantified using the average peak areas when compared with the mixture of standard (xylose, glucose, arabinose, acetic acid and furfural) and expressed as g/l sugar.

2.3. Ferulic acid

The determination of ferulic acid was carried out following the method developed by Saulnier et al. (1995). A total of 0.1 ml of the solution from the acid hydrolysate of wheat straw was mixed with a buffer of pH 10 (0.1 M sodium tetraborate – 0.1 M glycine buffer at pH 10). The amount of free and esterified ferulic acid were calculated from the absorptions (A) at 375 and 345 nm using molar absorption coefficients (ε_{345} = 19,662, ε_{375} = 7630 for free ferulic acid; and ε'_{345} = 23,064, ε' = 31,430 for esterified ferulic acid). The concentrations for both free $[FA]_f$ and esterified $[FA]_e$ ferulic acid were calculated using the following equations:

$$[FA]_e = \frac{[(A_{375} \times \varepsilon_{345}) - (A_{345} \times \varepsilon_{375})]}{[(\varepsilon'_{375} \times \varepsilon_{345}) - (\varepsilon'_{345} \times \varepsilon_{375})]}$$

$$[FA]_f = \frac{[A_{345} - (\varepsilon'_{345} \times [FA]_e)]}{[\varepsilon_{345}]}$$

2.4. Determination of total phenolic content

The phenolic content was measured by the Folin–Ciocalteu method (Singleton and Rossi, 1965) with slight modifications and expressed as grams of gallic acid equivalents. The samples, 0.1 ml and 2.3 ml of distilled water were mixed with 0.1 ml of Folin–Ciocalteu reagent and incubated for 8 min, followed by the addition of 1 ml of 70 g/l sodium carbonate solution with 2 ml of water. The mixture was allowed to stand for 2 h at room temperature before reading the absorbance at 750 nm.

2.5. Antioxidant activity

2.5.1. Ferric-reducing antioxidant power (FRAP)

Solutions of 300 mM acetate buffer (pH 3.6), 10 mmol/l TPTZ and 20 mmol/l $FeCl_3 \cdot 6H_2O$ were prepared, and fresh working solutions were used by mixing stock solutions in a ratio of 10:1:1 to prepare the FRAP reagent. The FRAP reagent (2900 μl) was mixed with 100 μl of the sample or standard, and the mixtures were kept at room temperature under dark for 30 min. The absorbance of ferrous tripyridyltriazine complex was measured at 593 nm with a spectrophotometer. A standard calibration curve was prepared using Trolox at a concentration range between 10 and 50 μM (Benzie and Strain, 1996).

2.5.2. Trolox equivalent antioxidant capacity (TEAC)

Solutions of 2,2′-azinobis (3-ethyl-benzothiazoline-6-sulfonic acid) (ABTS) (7 mM) and potassium persulfate (2.45 mM) were mixed and allowed to stand for 12–16 h to generate the ABTS 2,2′-azinobis (3-ethyl-benzothiazoline-6-sulfonate) radical cation ($ABTS^{\bullet+}$). The $ABTS^{\bullet+}$ stock solution was diluted with 20 mM sodium acetate buffer (pH 4.5) to obtain 0.7 absorbance reading at 734 nm. The antioxidant capacity was measured by mixing 2 ml of the sample with 0.2 ml of radical solution and by following the decline in the absorbance for 10 min with appropriate solvent blanks for each addition. The radical scavenging capacity was compared with that of Trolox and the results were expressed as TEAC values (mmol Trolox) (Re et al., 1999).

2.6. Experimental design and response surface methodology (RSM)

A 2^3 rotatable central composite design (CCD) was used to fit a second-order model. The design consisted of 20 sets of experiments. The quadratic model was selected for predicting the optimal point and expressed as:

$$Y = b_0 + b_1X_1 + b_2X_2 + b_3X_3 + b_{11}X_1^2 + b_{22}X_2^2 + b_{33}X_3^2 + b_{12}X_1X_2 + b_{13}X_1X_3 + b_{23}X_2X_3 \quad (1)$$

where Y represents the response variables (xylose and phenolic yield); b_0 is the interception coefficient; b_1, b_2, and b_3 are the linear terms; b_{11}, b_{22}, and b_{33} are the quadratic terms; and X_1, X_2, and X_3 represent the variables studied.

The Design Expert v. 7 (Stat-Ease Inc., Minneapolis) was used for regression and the graphical analyses of the data obtained. Fischer's test was used for the determination of the type of model equation, while the student's t-test was performed for the determination of statistical significance of regression coefficients.

3. Results and discussion

3.1. Sugar and byproducts

In the previous study, glucan (37%) was reported as a major component of wheat straw followed by xylan (23%) and lignin (klason lignin: 20% and acid-soluble lignin: 2%). The rest of its components were arabinan (2%), acetyl groups (1%), uronic acid (3%), protein (3%), ash (6%) and extractives (3%) (Akpinar et al., 2010a).

The concentrations of xylose and other degradation products showed dependence on the experimental operating conditions, which were also explained by the severity factor (R_0) (Fig. 1A). Severity factor combines the experimental effects of temperature, reaction time and acid concentration to make the comparison of the results easy (Kabel et al., 2007). The highest xylose yield was 0.169 g/g-wheat straw, achieved at 140 °C for 15 min with 2% acid concentration ($\text{Log}\,R_0$ = 1.13). During the hydrolysis of

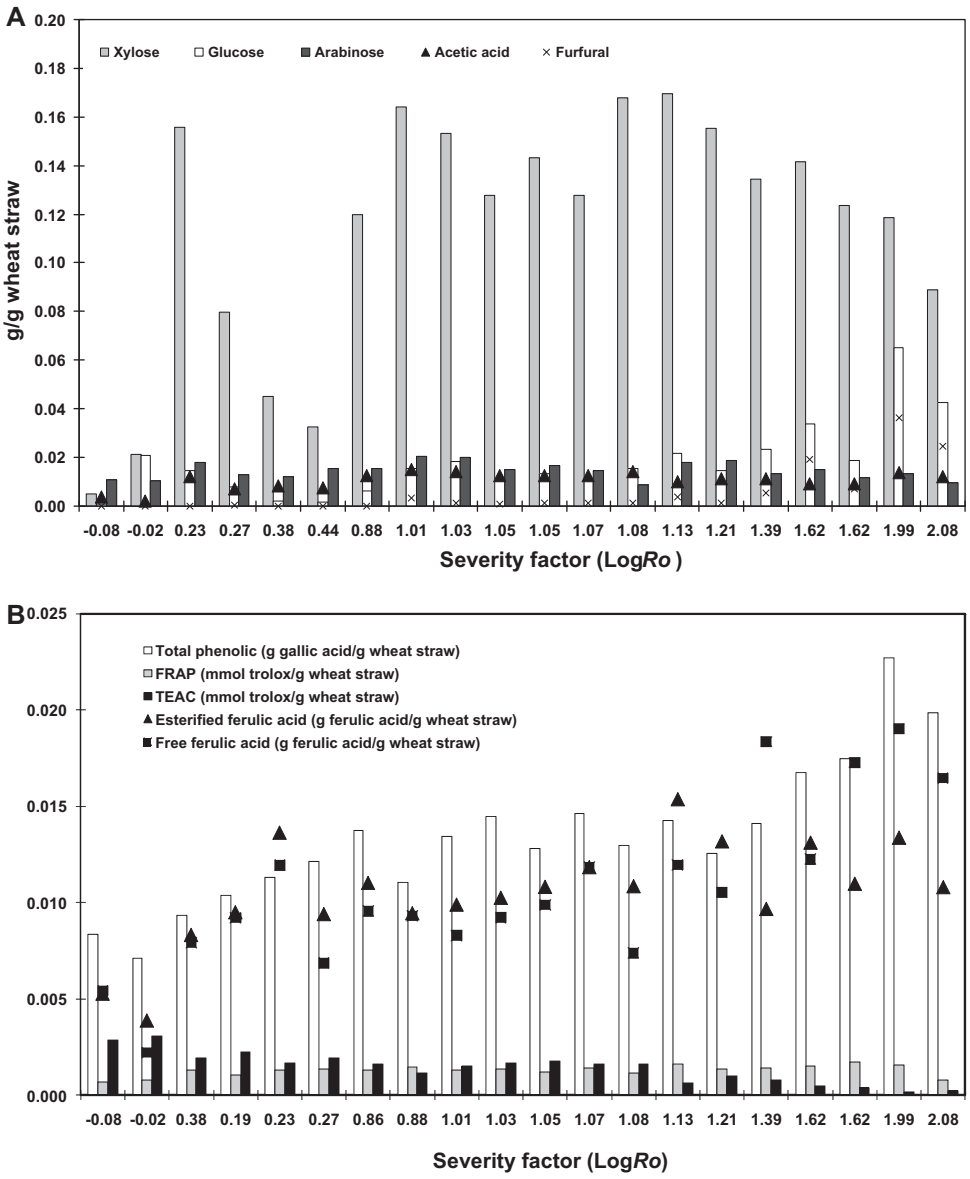


Fig. 1. (A) Formation of xylose, glucose, arabinose, acetic acid and furfural under selected hydrolysis conditions; (B) phenolic, free and esterified ferulic acid content, and antioxidant activity of acid hydrolysate under selected hydrolysis conditions. Severity factor: $R_0 = [10^{-pH} \times t \times \exp(T - 100)/14.75]$ of the pretreatments calculated from pH of hydrolysate.

agricultural wastes, other sugars, mainly glucose, were released, which were produced either from cellulosic fraction or from some heteropolymers of hemicellulosic fraction. The level of glucose in the fermentation media is important due to its the advers effect on xylose bioconversion to other chemicals such as xylitol (Roberto et al., 2003). In addition, during the production of xylose, another reaction takes place, which is the dehydration of xylose to furfural. Furfural formation not only decreases monosaccharide yield but also causes problems associated with the inhibition of fermentation of the sugars to, e.g., ethanol or xylitol (Karimia et al., 2006). When the operating temperature and reaction time were 153.3 °C and 30 min respectively and the acid concentration was maintained at 4% ($\text{Log}\,R_0 = 1.99$), glucose (0.063 g/g-wheat straw) and furfural (0.037 g/g-wheat straw) yields were maximum.

In all the experiments, arabinose yield remained between 0.01and 0.02 g/g-wheat straw and acetic acid yield was between 0.002 and 0.014 g/g-wheat straw. Low concentration of acetic acid in the hydrolysate is particularly important for xylose bioconversion because this acid is one of the potential inhibitors of the microbial metabolism (Roberto et al., 2003).

3.2. Antioxidant activity and phenolic content

The Folin–Ciocalteu test, used as a measure of total phenolics value in the hydrolysate, indicates the amount released from the extractives and lignin fraction during mild acid treatment. The ABTS scavenging test was selected because it is one of the widely used and simple techniques, and has been proposed as one of the methods considered for standardization of antioxidant activity in food and nutraceuticals (Prior et al., 2005). As the antioxidant capacity of a lignin fraction is related to its reducing ability, the ferric-reducing power was selected to measure the reducing capacity (Castro et al., 2008). The amounts of free and esterified ferulic acid were also evaluated in the acid hydrolysate of wheat straw because they have powerful antioxidant effect. It was reported that esterified ferulic acid shows higher antioxidant characteristics than free ferulic acid in the microsomal lipid peroxidation system and

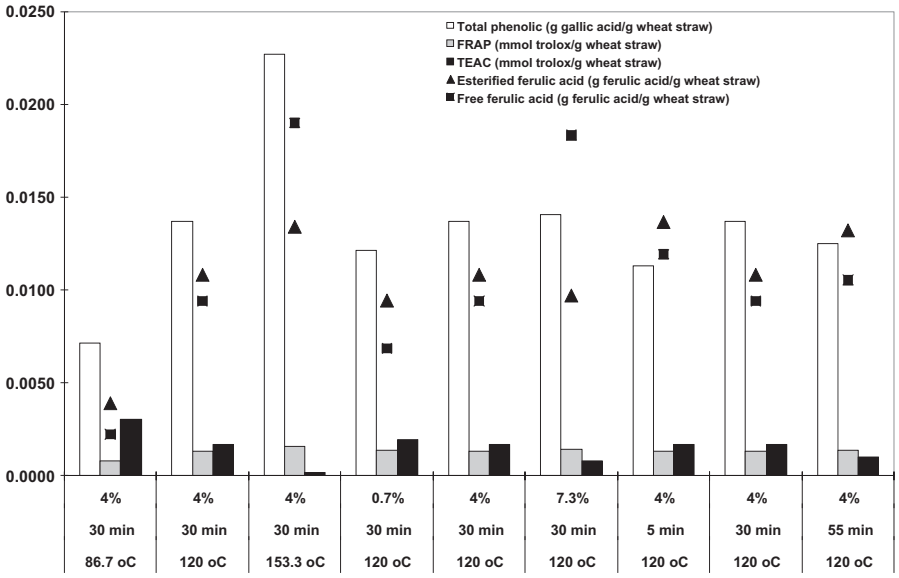


Fig. 2. The effect of temperature, time and acid concentration of penolic, free and esterified ferulic acid content, and antioxidant activity of acid hydrolysate.

Table 1
Experimental range and levels of independent process variables.

Independent variable	Symbol	Range and levels				
		$-\alpha$	−1	0	+1	$+\alpha$
Temperature (°C)	X_1	86.7	100	120	140	153.3
Reaction time (min)	X_2	5	15	30	45	55
Acid concentration (%)	X_3	0.7	2	4	6	7.3

Table 2
Experimental design and results obtained by hydrolysis of wheat straw and severity factor of the pretreatments.

Runs	Log R_0	Variables			Responses	
		X_1	X_2	X_3	Y_1 (g-xylose/g-wheat straw)	Y_2 (g-gallic acid/g-wheat straw)
1	−0.08	100	15	2	0.005	0.0084
2	−0.02	86.7	30	4	0.021	0.0071
3	0.19	100	45	2	0.033	0.0093
4	0.23	120	5	4	0.156	0.0104
5	0.27	120	30	0.7	0.080	0.0113
6	0.38	100	15	6	0.045	0.0121
7	0.86	120	30	4	0.168	0.0138
8	0.88	100	45	6	0.120	0.0110
9	1.01	120	30	4	0.128	0.0134
10	1.03	120	30	4	0.153	0.0145
11	1.05	120	30	4	0.164	0.0128
12	1.07	120	30	4	0.128	0.0146
13	1.08	120	30	4	0.143	0.0130
14	1.13	140	15	2	0.169	0.0143
15	1.21	120	55	4	0.155	0.0125
16	1.39	120	30	7.3	0.134	0.0141
17	1.62	140	45	2	0.124	0.0167
18	1.62	140	15	6	0.142	0.0175
19	1.99	153.3	30	4	0.119	0.0227
20	2.08	140	45	6	0.089	0.0199

Y_1, xylose yield (g-xylose/g-wheat straw); Y_2, total phenolic (g-gallic acid/g-wheat straw); R_0: severity factors ($R_0 = [10^{-pH} \times t \times \exp(T-100)/14.75]$).

Table 3
Analysis of variance for xylose yield and total phenolics content.

Source	Sum of squares		Degress of freedom		Mean square		F-value		P-value	
	Y_1	Y_2	Y_1	Y_2	Y_1	Y_2	Y_1	Y_2	Y_1	Y_2
Model	0.046	0.00025	7	5	0.0066	0.00005	23.03	27.36	<0.0001	<0.0001
Residual	0.0034	0.000014	12	14	0.00029	0.000001				
Lack of fit	0.0019	0.000012	7	9	0.00028	0.0000013	0.91	2.62	0.5597	0.1913
Pure error	0.0015	0.0000028	5	5	0.00030	0.00000057				
Total	0.05	0.00026	19	19						
R^2	0.93	0.95								

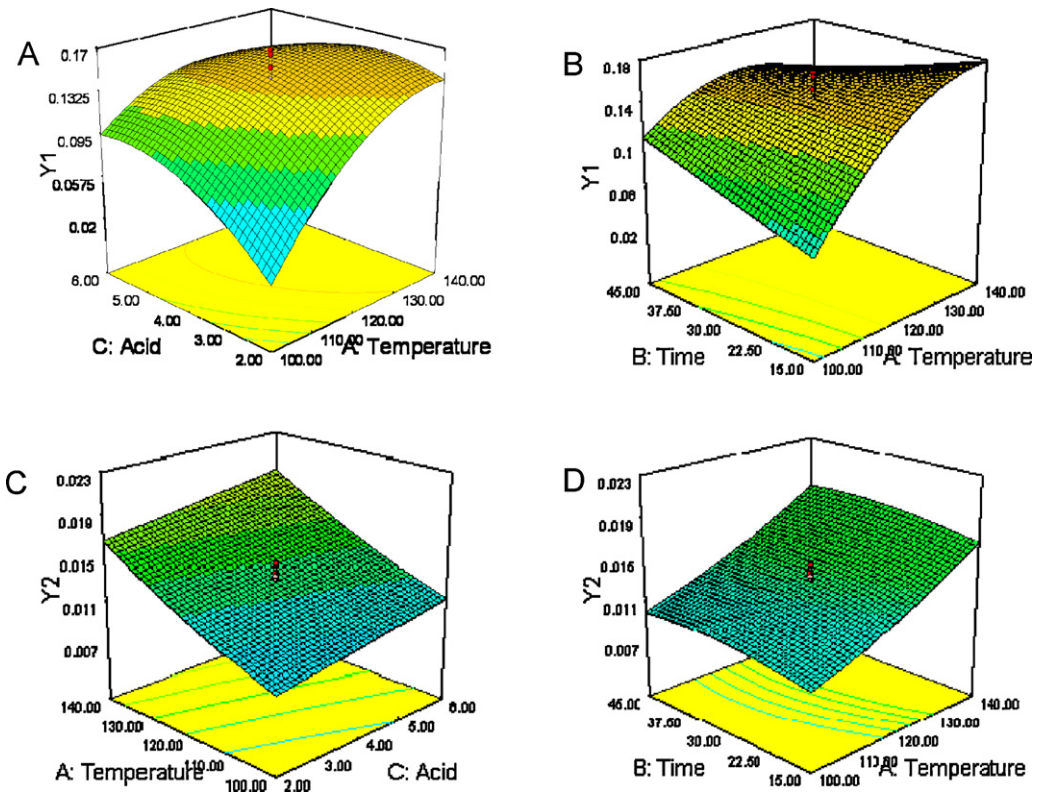


Fig. 3. (A) Effect of H_2SO_4 concentration and reaction temperature on xylose yield when time was set at 30 min as the center point. (B) Effect of reaction temperature and time on xylose yield when acid concentration was set at 4% as the center point. (C) Effect of H_2SO_4 concentration and reaction temperature on phenolic content when time was set at 30 min as the center point. (D) Effect of reaction temperature and time on phenolic content when acid concentration was set at 4% as the center point.

reduced the oxidation of low-density lipoproteins (Li et al., 2008).

The most active radical scavenger of wheat straw hydrolysate was generated at 153.3 °C for 30 min with 4% acid concentration. The redox potential of Fe^{3+} salts used in the FRAP assay was comparable with that of the TEAC value, and correlation with phenolic content was observed (Fig. 1B). Esterified ferulic acid was highest at 140 °C for 15 min and 2% acid concentration (Log R_0 = 1.13), and its amount was higher than free ferulic acid. When the severity of the operation conditions was increased, the amount of free ferulic acid started to increase and the value became higher than the esterified one.

The yield in phenolic compound, including antioxidant activity and free and esterified ferulic acid, was not affected significantly by the sulfuric acid concentration and the reaction time used in the acid hydrolysis stage, but was strongly affected by the temperature. The highest phenolic content was 0.0227 g-gallic acid/g-wheat straw, achieved at 153.3 °C for 30 min with 4% acid concentration (Log R_0 = 1.99) (Fig. 2).

3.3. Statistical modeling

The experimental range and levels of independent variables investigated are given in Table 1. The design of this research, including the dependent variables or responses, xylose yield (Y_1) and phenolic content (Y_2), is given in Table 2. The quadratic models with coded variables are shown in Eqs. (2) and (3), which represent the xylose yield (Y_1) and phenolic content (Y_2) as a function of temperature (X_1), time (X_2) and acid concentration (X_3).

$$Y_1 = 0.15 + 0.035X_1 + 0.00026X_2 + 0.012X_3 - 0.03X_1^2 - 0.023X_3^2 - 0.025X_1X_2 - 0.024X_1X_3 \quad (2)$$

$$Y_2 = 0.013 + 0.0039X_1 + 0.00060X_2 + 0.0013X_3 + 0.00057X_1^2 - 0.00064X_2^2 \quad (3)$$

Regression analysis was performed to fit the response function and experimental data. The second-order models for xylose and phenolic yield were evaluated by ANOVA, which are shown in Table 3. For both responses, the regression was statistically significant at 95% confidence level. The model for both responses did not show lack of fit. The determination coefficient (R^2) for the first (Y_1) and the second response (Y_2) was 0.93 and 0.95 respectively, explaining the 93 and 95% of variability in the responses.

Fig. 3 shows the response surfaces to estimate the xylose and phenolic yield over the independent variables of temperature (X_1), time (X_2) and acid concentration (X_3). When the reaction time was set at 30 min as center point, as shown in Fig. 3A and C, it was interpreted that the maximum xylose yield (0.16 g/g-wheat straw) was obtained with a 3.8% acid concentration and 129 °C reaction temperature. The maximum phenolic content was 0.019 g-gallic acid/g-wheat straw at 6% acid concentration with 140 °C reaction temperature. Fig. 3B and D shows the effect of temperature and time on xylose yield and the phenolic content of the hydrolysate when the reaction time was set acid concentration was 4% as the center point. The maximum xylose yield (0.18 g/g-wheat straw) was obtained at 140 °C and 15 min reaction time, and the maximum phenolic content (0.018 g-gallic acid/g-wheat straw) of the hydrolysate was obtained at 140 °C and 45 min reaction time.

When the overall xylose yield and phenolic content of the acid hydrolysate of wheat straw were compared with those obtained from previous studies carried out for the production of xylose (Roberto et al., 2003; Akpinar et al., 2010b) and phenolic compounds (Moure et al., 2008) from lignocellulosic materials with acid hydrolysis, it was found that the xylose yield and the phenolic contents were similar. The results showed that increase in severity of

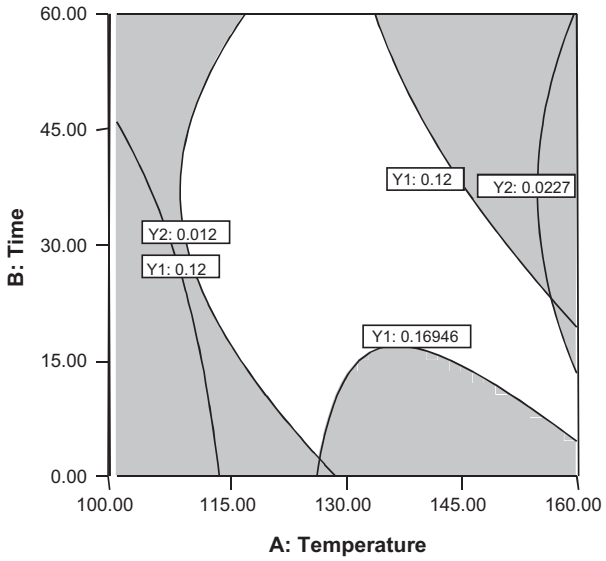


Fig. 4. Overlaying plots of xylose yield and phenolic content of the acid hydrolysate of wheat straw.

the hydrolysis conditions of wheat straw resulted with increase in phenolic yields and decrease in xylose yield.

Based on the two models, a graphical optimization that consisted of overlaying the contour plots of both the models was conducted. The optimal working conditions based on high level xylose yield and phenolic content were chosen using the following criteria: xylose yield > 0.12 g/g-wheat straw and phenolic content > 0.012 g-gallic acid/g-wheat straw. In the overlaying plot (Fig. 4), the regions with a shaded area do not fit the optimization criteria, while the non-shaded area meets the optimization criteria. As an optimum point, 4.7% acid concentration, 120 °C and 45 min were selected. Under this condition, the severity factor of the treatment, calculated as Log R_0, was found to be 1.2. Under these conditions, the xylose yield and phenolic content were predicted as 0.15 g/g-wheat straw and 0.014 g-gallic acid/g-wheat straw, respectively. To confirm these results, hydrolysis runs were conducted under these optimized conditions, the xylose and phenolic yield of wheat straw were obtained as 0.16 g/g-wheat straw and 0.014 g-gallic acid/g-wheat straw, respectively.

4. Conclusion

Higher severities of conditions resulted in improved yields of phenolic content of the acid hyrolysate of wheat straw, but in decreased xylose yield. The optimum reaction conditions were found as 4.7% acid concentration, 120 °C and 45 min for wheat straw. Under selected hydrolysis conditions, wheat straw proved to be a promising source of high yield xylose, which could be used for the production of different chemicals, mainly xylitol and phenolic compounds which exhibited high antioxidant activities. The results showed that wheat straw had the potential applications area for the polysaccharide-based antioxidants in food, pharmacy and cosmetics.

Acknowledgment

This work was partially supported by The Scientific and Technological Research Council of Turkey.

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