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SHORT COMMUNICATION

Optimization of sono-assisted dilute sulfuric acid process for simultaneous pretreatment and saccharification of rice straw

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Abstract In this study, sono-assisted dilute sulfuric acid process was evaluated for its viability of simultaneous pretreatment and saccharification of rice straw. Three critical factors for simultaneous pretreatment and saccharification process, such as sonication time (30–50 min), temperature (70–90 °C), and acid concentration (5–10 %), were optimized to maximize reducing sugar yield using Box-Behnken design and response surface methodology. The response surface methodology model was found to be adequately fitted to the obtained data. Simultaneous pretreatment and saccharification factors were optimized at sonication of 50 min, 80 °C and an acid concentration of 10 % yielding the maximum sugar content (31.78 g/100 g of biomass). Scanning electron microscopy revealed that the smooth surface of raw biomass was altered into a rough and porous surface as a result of sugar release, which showed the prospective feasibility of simultaneous pretreatment and saccharification process. This process integration may lead to develop economical bioethanol production facility. However, further research is required to make this process industrially viable.

Keywords Bioethanol · Lignocellulosic biomass · Process integration · Ultrasound

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Introduction

Alternatively, renewable energy options have been the global priority since the last decade due to the risks of global warming and energy security. Biofuels have appeared one of the most viable and green alternatives among various renewable energy sources (Nigam and Singh 2010). First- and second-generation biofuels are derived either from crops such as corn, sugarcane, or from various nonfood lignocellulosic biomasses. The utilization of food crops as feedstock can raise the price of these commodities and can potentially lead to the risks of food insecurity (Man et al. 2010; Shen et al. 2011). Lignocellulosic biomass (LCB) can alternatively be used as a sustainable and cheap feedstock for the production of bioethanol which will not only facilitate agricultural residue management, but it will also provide fuel for various sectors of any economy. This decentralized approach will assist in developing rural economy, and it will lower the energy import bill. Although bioethanol derived from LCB offers numerous advantages, yet its commercial production is hindered by some economic and technical obstacles (Alvira et al. 2010; Baboukani et al. 2012).

The production chain of lignocellulosic bioethanol is comprised of following main steps: pretreatment, hydrolysis, and fermentation (Conde-Mejía et al. 2012). LCB inherits a recalcitrant structure and complex arrangement of polysaccharides. This situation requires such a pretreatment method which can make polysaccharides accessible for the release of fermentable sugars for downstream processing (Baboukani et al. 2012; Ribeiro et al. 2012; Xu et al. 2011). Several pretreatment techniques have been successfully employed including physical, biological, chemical, and their combinations. These pretreatment technologies either employ harsh process conditions in

terms of temperature, pressure, and chemicals (steam explosion, dilute acid/alkali pretreatment) or require long processing time (biological pretreatment). No standalone pretreatment option is available yet for commercial scale applications (Alvira et al. 2010). Besides pretreatment, hydrolysis of pretreated LCB to fermentable sugars is another bottleneck because several factors affect the digestibility of these sugars. Hydrolysis of pretreated LCB can be accomplished either by acid hydrolysis or enzymatic hydrolysis. Enzymatic hydrolysis is associated with high cost due to the utilization of pure enzymes (Chandel et al. 2007). Thus, pretreatment and hydrolysis of LCB both appear as a cost-limiting step in the production chain of bioethanol.

Acid catalyzed (mainly sulfuric acid) pretreatment has been extensively investigated to pretreat several LCBs (Yaqoob et al. 2012). It effectively solubilizes hemicellulose and alters the biomass structure by interacting with bonding of biomass components (Limyem and Ricke 2012). Dilute acid (DA) is normally preferred over concentrated acid due to its cost effectiveness and environment-friendly character. DA can also catalyze disruption of glucosidic bonds between sugar monomers within LCB in the hydrolysis step (Aguilar et al. 2002). Grethlein and Converse (1991) reported that the hydrated hydrogen ions can access glucosidic bonds easily and equally even in the case of different LCBs than the cellulase enzyme which make DA hydrolysis more efficient (Baboukani et al. 2012). Thus, DA application can be extended from pretreatment to hydrolysis step targeting total hydrolysis of LCB to fermentable sugar monomers. However, DA pretreatment requires high temperature (>100 °C) for a longer period of time (30–90 min) which can degrade released sugars to produce some inhibitory compounds (Alvira et al. 2010). High temperature and longer reaction time can be avoided by integrating DA with some suitable thermo-mechanical technique such as ultrasonication.

Ultrasonic waves (20 kHz) are applied to LCB in liquid suspensions where energy is simultaneously provided following a complex chemistry in the form of cavitation, agitation, turbulence, and heating (Vilkhu et al. 2008; Fernández-Cegrí et al. 2012). Hydro-mechanical shear forces are produced in the bulk liquid due to cavitation which increases mass transfer at the solid–liquid interface, and ultimately, mechanical energy leads to particle size reduction of exposed material (Velmurugan and Muthukumar 2012; Rehman et al. 2013). Thus, it can be effectively employed to pretreat LCB for subsequent processing (Yang et al. 2012). This combinational energy can offer application of sonication even at mild process conditions. Sonication has been effectively employed for pretreatment (Harun et al. 2011; Velmurugan and Muthukumar 2011, 2012; Yang et al. 2012) as well as DA hydrolysis (Velmurugan and Muthukumar 2011) and enzymatic hydrolysis of LCB (Yachmenev et al. 2009; Velmurugan and Muthukumar 2012) at mild temperature conditions (25–50 °C). However, ultrasonication was used either single or coupled with other techniques during pretreatment and hydrolysis steps. Those published arts motivated us to investigate any synergetic benefit of sono-assited DA process for process intensification. Both ultrasonication and DA have been successfully used for pretreatment and hydrolysis of LCBs; thus, we hypothesize that if ultrasonication is coupled with DA it will not only pretreat the LCB but also hydrolyze the biomass simultaneously in one single step (Nugraha et al. 2010). This process integration would remove one complete step from the entire bioethanol production chain leading to the improved process economics. Therefore, the primary objective was to examine the viability of sono-assisted DA technique as a simultaneous pretreatment and saccharification (SPS) process and to optimize process conditions such as sonication time, temperature, and acid concentration using response surface methodology (RSM) to maximize the sugar yield. This study was carried out in January 2012 at Korea Advanced Institute of Science and Technology, Republic of Korea.

Materials and methods

Material

Rice straw (RS) was obtained from a local field in Daejeon, South Korea. The biomass was milled using lab scale grinder, and the ground RS was screened to achieve the particle size <3 mm. The screened RS was air-dried at 45 °C prior to the pretreatment. RS compositional analysis showed that it contained 36.5 % glucan, 20.8 % xylan, and 16.9 % lignin on dry-weight basis according to the Lab Analytical Procedure (LAP) developed by National Renewable Energy Laboratory (NREL), USA (Sluiter et al. 2006).

Simultaneous pretreatment and saccharification of RS

The simultaneous pretreatment and saccharification of RS were carried out in a Teflon-coated stainless steel reactor (70 mL). The reactor was placed in an oil bath to undertake the experimentation at desired temperature levels. A probe type ultrasonic processor (VCX750, Sonics & Materials Inc, USA) with an operating frequency of 20 kHz, power 750 W, was used employing 20 % of amplitude. The ultrasonic processor probe was submerged in the center of reactor at maximum in such a way that it did not touch either the bottom or walls of the reactor to ensure uniform irradiations on the reaction mixture (Harun et al. 2011).

Table 1 Actual and coded level of factors tested with Box–Behnken design

Code	Factor	Unit	−1	0	1
A	Sonication time	min	30	40	50
B	Temperature	C	70	80	90
C	Acid Conc.	%	5	7.5	10

The experimental conditions (sonication time, temperature, and acid concentration) were maintained in accordance with the experimental design given in the subsequent section. A liquid to solid ratio of 10 was employed for all the experimental runs. The reactor was immediately placed in cold water after each run to stop any further reaction. The reaction mixture was withdrawn from the reactor once it attained room temperature, and it was filtered. The filtrate was subjected to sugar analysis according to LAP-NREL (Sluiter et al. 2006). The residue was subject to scanning electron microscopy to notice structural changes in the SPS-treated biomass.

Experimental design and statistical optimization

Sonication time, temperature, and acid concentration were selected as experimental factors in this study. The effect of these independent factors on the reducing sugar yield (response) was investigated employing Box-Behnken design (BBD) of response surface methodology. Three independent factors, sonication time (A), temperature (B), and acid concentration (C), were studied at the three levels (−1, 0, +1). The range and levels of these factors are presented in Table 1. The experimental design matrix is given in Table 2. The amount of reducing sugars, glucose, and xylose mainly was selected as the response vector, Y (g of sugar/100 g of dry RS). Experimental data were analyzed using regression analysis employing a quadratic model as shown in the given equation:

$$\mathrm{Y}\ (\mathrm{g}/100\mathrm{g}) = \beta_{\mathrm{o}} + \beta_1\mathrm{A} + \beta_2\mathrm{B} + \beta_3\mathrm{C} + \beta_4\mathrm{AB} + \beta_5\mathrm{AC} + \beta_6\mathrm{BC} + \beta_7\mathrm{A}^2 + \beta_8\mathrm{B}^2 + \beta_9\mathrm{C}^2 \quad (1)$$

where β's are the regression coefficients for intercept, linear, quadratic, and interaction terms. The experimental design and its analysis were carried out by Design-Expert 8.0.7.1 Trial version (State-Ease, Inc., Minneapolis, MN, USA).

Results and discussion

Several studies have reported that the pretreatment and hydrolysis of biomass are influenced by the factors including reaction time, temperature, and acid concentration (Kim et al. 2011). Some initial experiments (based on literature review and lab studies in our research group) were carried out in order to determine the levels of selected factors prior to optimization using RSM (Velmurugan and Muthukumar 2011). It was observed, on the basis of preliminary experiments, that sono-assisted dilute acid process could simultaneously couple pretreatment and saccharification process under mild process conditions (data not shown). The levels of selected factors are given in Table 1, whereas BBD experimental design matrix and results have been presented in Table 2. Quadratic equation used for the model is given below:

$$\mathrm{Y}_{\mathrm{sugar\ yield}}(\mathrm{g}/100\,\mathrm{g}) = 25.67 + 2.21\mathrm{A} + 5.12\mathrm{B} + 2.69\mathrm{C} - 0.73\mathrm{AB} + 1.42\mathrm{AC} - 0.63\mathrm{BC} - 0.97\mathrm{A}^2 - 2.13\mathrm{B}^2 - 1.59\mathrm{C}^2 \quad (2)$$

where A, B, and C are the coded values of sonication time, temperature, and acid concentration, respectively. The factors, having positive values for their coefficients, improved the response when the level of those factors was raised, whereas negative value of coefficients suggested their inverse relationship with the response vector. Figure 1a shows the correlation between experimental and predicted sugar yield ($R^2 = 0.909$; $R^2_{\mathrm{adj}} = 0.745$; $\%\mathrm{CV} = 11.3$). Figure 1b reveals that the residuals are normally distributed against experimental sugar yield implying the fitness of RSM model (Cruz-González et al. 2012; Han et al. 2011a, b; Qi et al. 2009). Thus, reducing sugar yield and response surface curves could be satisfactorily predicted using Eq. 2. Furthermore, Baboukani et al. (2012) suggested criteria to ensure model fitness. According to this criteria, $R^2 > 0.80$ and the difference between the values of R^2 (0.909) and R^2_{adj} (0.745) should not exceed by 0.20. The results of present study met the suggested criteria. However, predicted yield was slightly different from the experimental values (Table 2) in the range of ±2.6 due to some lack of fitness (Córdova et al. 2011). The analysis of variance (ANOVA) of RSM model is given in Table 3. The terms with $p < 0.05$ are statistically significant at 95 % confidence level. Temperature and acid concentration were found statistically significant, whereas sonication time was nonsignificant. No significant interaction (AB, AC, and BC) among the tested factors was observed ($p \geq 0.328$). Similarly, quadratic terms (A^2, B^2, and C^2) did not show any significance ($p \geq 0.18$). Thus, linear effects (A, B, and C) were more significant than their interactions and quadratic terms. Man et al. (2010) and Khalili et al. (2011) have reported similar observations in the literature. All of the interactions and quadratic terms showed negative effect on the sugar yield except AC.

Table 2 Box-Behnken design matrix for optimization of factors and the response values for yields of total reducing sugar produced, predicted and their residuals

Run	Coded variable levels			Reducing sugar (g/100 g)		
	A-Time (min)	B-Temperature (°C)	C-Acid conc. (%)	Experimental	Predicted	Residual
1	40	80	7.5	25.67	25.67	0.003
2	30	80	10	23.61	22.17	−1.44
3	40	90	5	27.61	25.01	−2.60
4	40	80	7.5	25.67	25.67	0.00
5	40	70	5	14.68	13.51	−1.17
6	50	70	7.5	20.66	20.39	−0.27
7	40	80	7.5	25.67	25.67	0.001
8	30	80	5	17.28	19.63	2.35
9	50	80	10	31.78	29.43	−2.35
10	30	70	7.5	15.70	14.51	−1.19
11	50	90	7.5	27.98	29.17	1.18
12	30	90	7.5	25.93	26.21	0.27
13	50	80	5	19.77	21.21	1.43
14	40	70	10	17.54	20.15	2.60
15	40	90	10	27.97	29.13	1.16

Although, sonication time did not appear significant as an individual effect, but its interaction with acid concentration (AC) was significant to improve the sugar release from rice straw. Besides their non-significance, interaction, and quadratic terms were not omitted from the quadratic model as suggested in some studies (Chauhan and Gupta 2004; Han et al. 2011a) because the quadratic and interaction terms (even non-significant) contribute to the response vector up to certain extent (Khalili et al. 2011).

Three-dimensional response surface plots were constructed to find out the optimum level of each factor with an objective to maximize the sugar yield. Response surface plots estimate sugar yield based on two factors at a time while remaining factor is fixed at its central level. Graphic representation of these response surfaces is presented in Figs. 2, 3, and 4. Figure 2 shows the effect of sonication time and temperature on the yield of reducing sugars produced at a fixed acid concentration of 7.5 %. The sugar yield was twice when the sonication time was increased from 30 to 50 min. The maximum yield of 31.78 g/100 g reducing sugars was recovered after 50 min of sonication. Researchers had also reported the increased sugar yield as a function of sonication time (Harun et al. 2011; Velmurugan and Muthukumar 2011, 2012; Yunus et al. 2010).

The interaction between sonication time and acid concentration is shown in Fig. 3 at a fixed temperature of 80 °C. Sugar yield gradually increased with the increase in sonication time and acid concentration. The maximum amount of reducing sugars was achieved at 50 min of sonication and 10 % sulfuric acid concentration. Yunus et al. (2010) had reported that the combination of

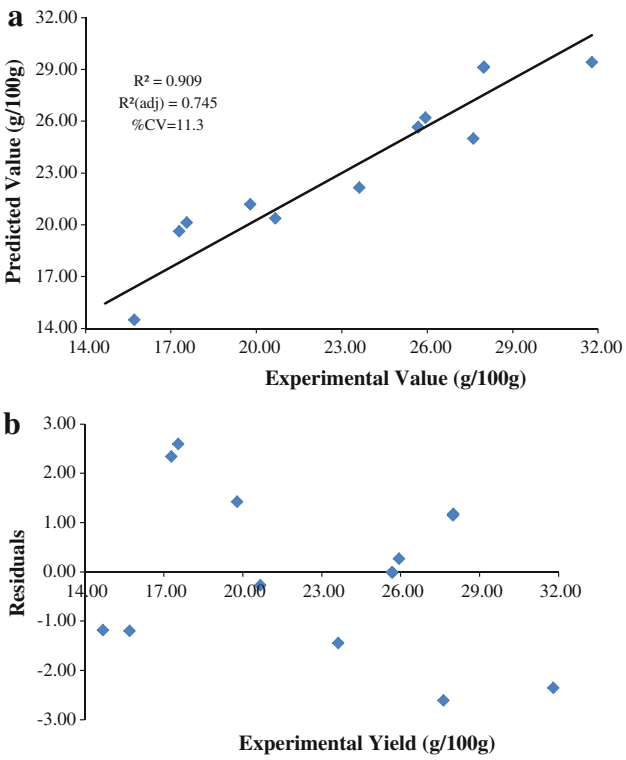

Fig. 1 Fitness of RSM model. **a** correlation between experimental and predicted sugar yield. **b** distribution of residual against experimental sugar yield

sonication time and acid hydrolysis at elevated temperature showed a positive impact on the yield of reducing sugar. The sugar yield proved a linear function of sonication time and acid concentration. The effect of temperature and acid

Table 3 ANOVA for total reducing sugar produced (Y) as a function of sonication time (A), temperature (B), acid conc. (C) used during SPS process of RS

Source	Sum of squares	*df*	Mean square	*F* value	Prob > *F*
Model	343.902	9	38.211	5.543	0.036
A-Sonication time	39.028	1	39.028	5.662	0.063
B-Temperature	209.203	1	209.203	30.350	0.002
C-Acid Conc	58.104	1	58.104	8.429	0.033
AB	2.117	1	2.117	0.307	0.603
AC	8.065	1	8.065	1.170	0.328
BC	1.562	1	1.562	0.226	0.654
A^2	3.399	1	3.3998	0.493	0.513
B^2	16.588	1	16.588	2.406	0.181
C^2	9.183	1	9.183	1.332	0.300
Residual	34.464	5	6.892	–	–
Lack of fit	34.461	3	11.487	–	–

concentration is given in Fig. 4 (sonication time = 40 min). It was noticed that maximum yield was obtained at 10 % sulfuric acid concentration and 80 °C. The interaction was quadratic in nature because central level of temperature (80 °C) produced maximum sugar yield (31.78 g/100 g) at a fixed sonication time. The results of response surface indicated that all the factors in general had exhibited a linear and synergetic effect on the sugar yield as revealed earlier by ANOVA. Lee et al. (1999) anticipated that sugar yield could be maximized in batch process based on the ratio of rate constants depending on the levels of acid concentration and temperature.

The factor levels optimized by the RSM model were as follows: sonication time: 50 min; temperature: 90 °C; and acid concentration: 10 %. These factor levels represented maximum values as given in Table 1. These optimal levels were probably suggested by the model due to linearity observed in the response with respect to these factors as displayed in Figs. 2, 3, and 4. The quadratic model (Eq. 2) predicted a sugar yield of 31.068 g/100 g against the optimized conditions. Experiments were run in duplicate at optimized factor levels to confirm the validity of RSM model. An experimental yield of 31.425 g/100 g ($n = 2$) was obtained with a residual of −0.36 g/100 g, and 1 % error. This result further verified the adequacy of the RSM model. Although the optimization results showed the fitness of RSM model, but sugar yield did not increase as desired. The sugar yield, at factor levels suggested by RSM model, was lower than that obtained at 50 min of sonication time, 80 °C temperature, and 10 % acid concentration (Run 9, Table 2). In order to meet our objective of maximizing sugar yield in SPS process, factor levels were considered optimized against the maximum yield achieved. This option produced maximum yield even at lower temperature (80 °C than 90 °C). This decision was justified by the fact that lower temperature level (by 10 °C) could make the process more economical at industrial scale in terms of energy requirement and sugar yield. Velmurugan and

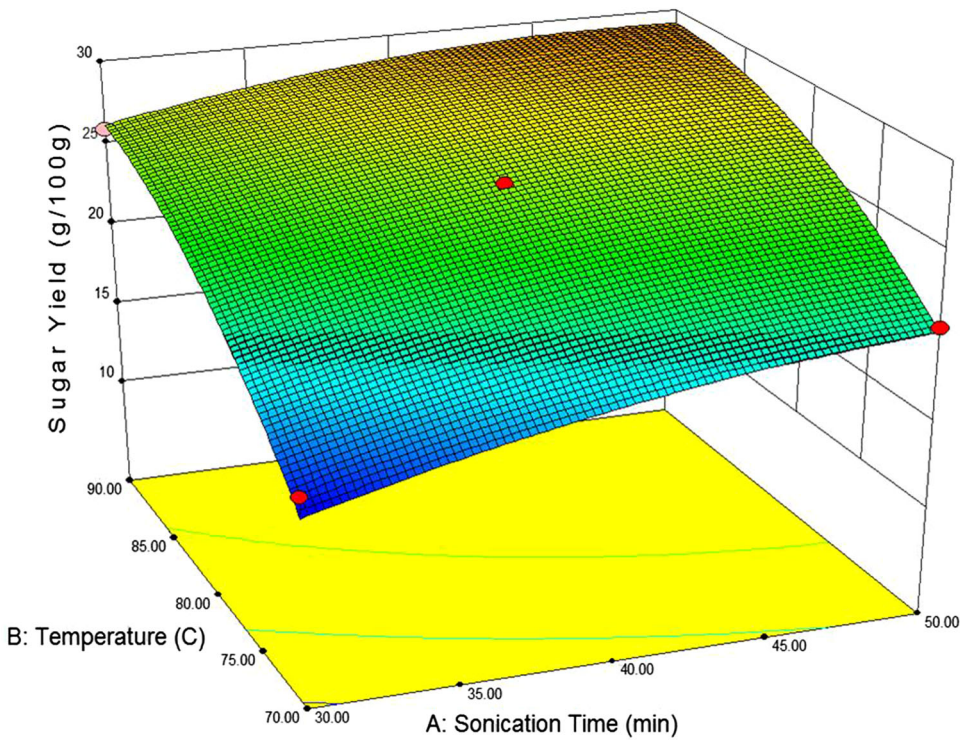

Fig. 2 Effect of sonication time and temperature on reducing sugar yield

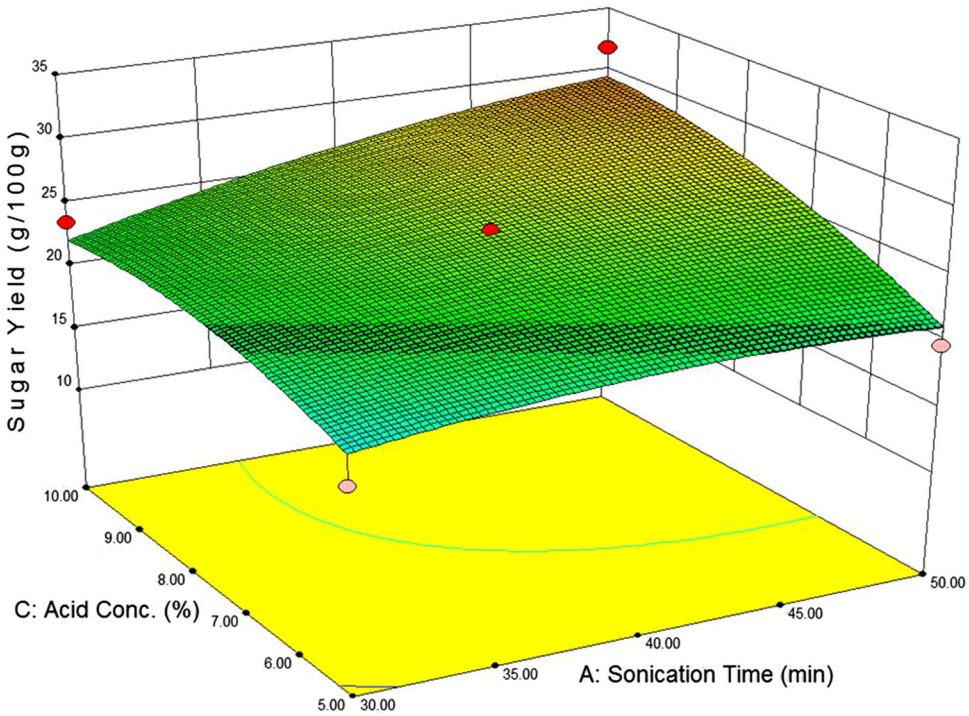

Fig. 3 Effect of sonication time and acid concentration on reducing sugar yield

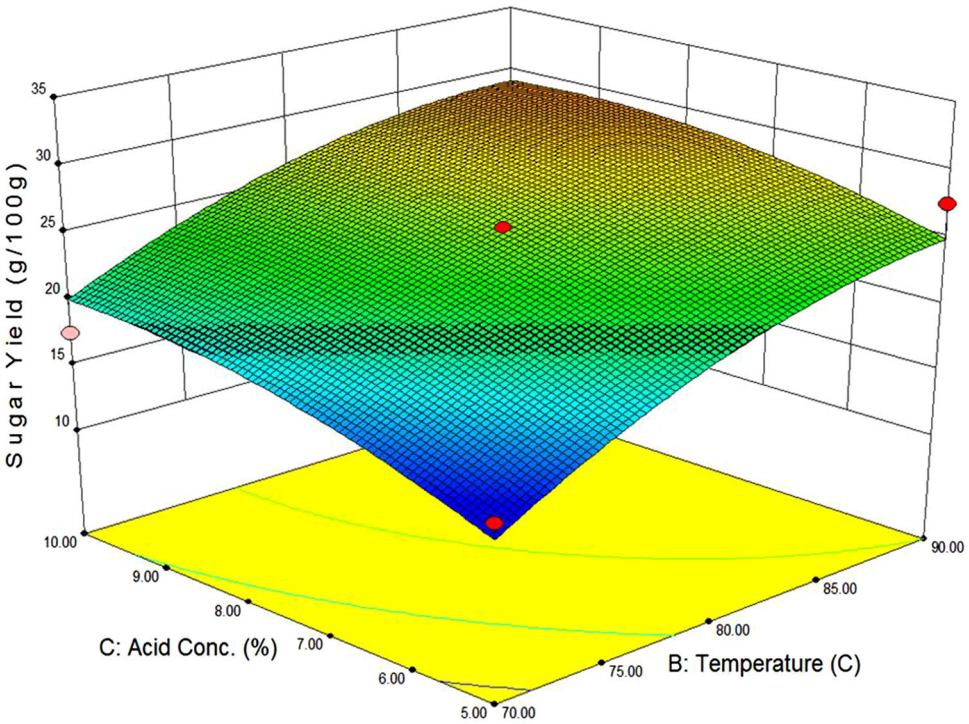

Fig. 4 Effect of temperature and acid concentration on reducing sugar yield

Muthukumar (2011) have reported a sugar yield of 28 g/L after sono-assisted acid hydrolysis which is comparable with our yield. However, biomass was first subjected to sono-assisted alkali pretreatment and was then hydrolyzed employing sono-assisted DA hydrolysis process. The duration of sonication spanned of 65 min (versus 50 min in this study). Moreover, pH adjustment between pretreatment and hydrolysis steps could have required some additional costs and resources. Harun et al. (2011) reported a sugar yield of 13.3 g/100 g as a maximum value where biomass was sonicated for 20 min initially, and pretreated biomass was further acid hydrolyzed at 121 °C for 60 min. Thus,

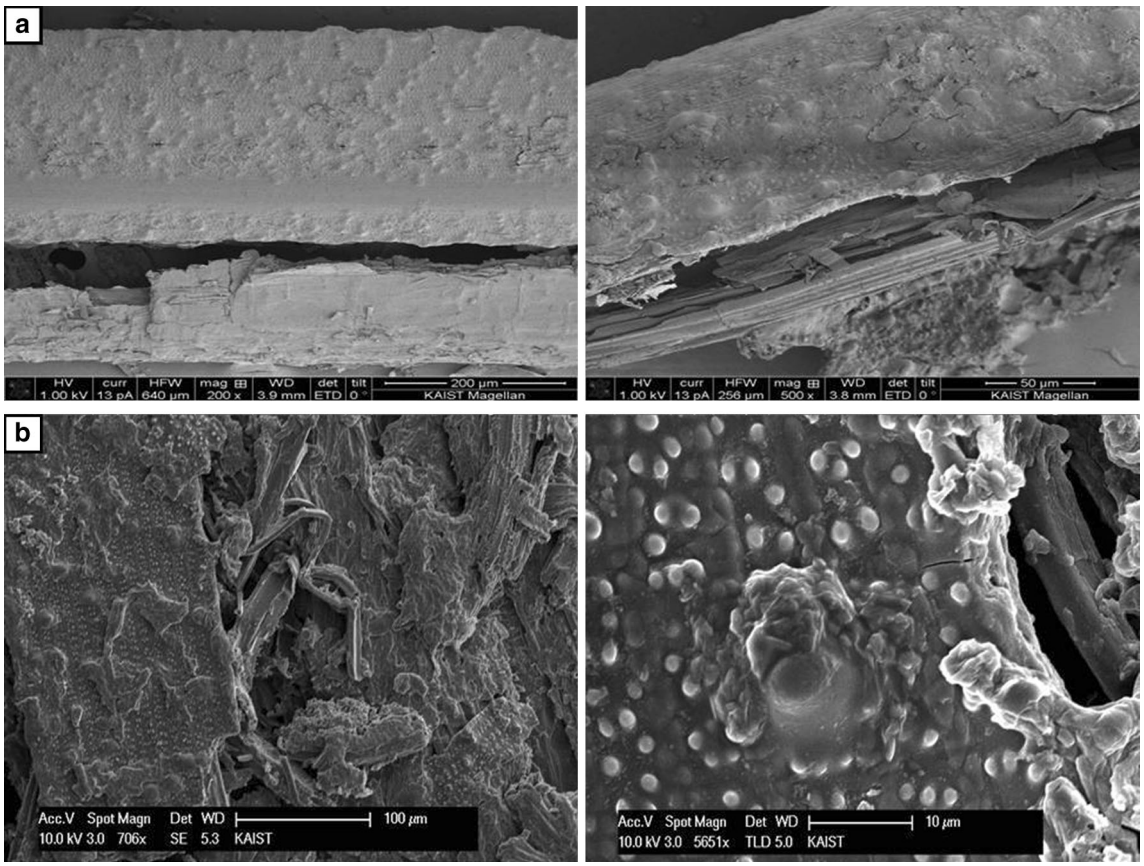

Fig. 5 Scanning electron micrograph: **a** raw RS **b** SPS treated at 90 °C, 50 min sonication, 10 % DA

less than half of the yield was obtained even after 80 min employing higher temperature compared with the present study.

The effect of SPS process was envisaged to improve the sugar yield. Hence, it was vital to investigate the morphological changes in RS biomass to correlate the higher sugar yield with the SPS process. Raw and treated RS biomass samples were observed under scanning electron microscope (SEM) to notice possible change in their morphologies. Scanning electron micrographs of untreated and SPS-treated RS are shown in Fig. 5. The raw rice biomass had a regular, compact, and smooth surface; whereas SPS-treated biomass displayed an irregular and rough surface. SPS process under the synergetic influence of sonication, dilute acid, and temperature facilitated the cleavage of RS structure. Micro-bubbles of dilute acid generated by sonication produced physio-chemical impact on the external surface which created micro-porous surface. These pores allowed the penetration of dilute acid micro-bubbles making the interior of biomass more accessible. This fact was proved by the solubilization and liberation of reducing sugars (Chukwumah et al. 2009; Yunus et al. 2010; Harun et al. 2011). Similar findings had been reported in the literature (Qi et al. 2009; Kim et al. 2011; Velmurugan and Muthukumar 2011, 2012) for sugarcane, barley straw, and Chinese white poplar fiber biomass treated by sulfuric acid and sonication.

Conclusion

The present study proposed a new process which could simultaneously pretreat and hydrolyze rice straw with higher reducing sugar yield. SPS produced 31.78 g of sugar/100 g of dry biomass employing sonication time of 50 min, 80 °C and 10 % sulfuric acid. SPS process led to the elimination of one complete processing step from the bioethanol production chain which could significantly improve the process economics. Further investigations regarding acid recovery and alternate source of sonication can even lower the operational cost.

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