Microwave and Steam Mediated Alkaline Pretreatments of Sweet Sorghum Bagasse: Study of the Energy Efficiency

Teodor Vintila, Radu Sumalan,* Nicolae Florin Popa, Monica Dragomirescu, and Florin Sala

The effects of microwave and steam mediated pretreatments were evaluated and compared relative to the hydrolysis of sweet sorghum bagasse (SSB) in the presence of sodium hydroxide. The results established the optimum parameters for energy consumption and sugar production after hydrolysis of the pretreated SSB. The energy balance was better in microwave treatment compared with the steam treatment. The results recommended pretreatment at 300 watts power setting in the microwave oven up to 10 min. At higher wattage setting and longer exposure time, increased sugar yields were obtained, although the efficiency of the process decreased when the energy balance was calculated. The energy balance was calculated as the proportion of energy consumed during the pretreatments to the energy that can be produced by fermenting released glucose to ethanol. In this study, the energy consumed during the different pretreatments varied from 1/33 to 1/200 of the energy obtainable by fermenting released glucose to ethanol. Overall, the pretreatment of SSB to produce fermentable sugars was effective, and the energy balance was positive both in microwave and steam pretreatments of alkaline suspension of SSB. In microwave treatment the energy applied can be more accurately dosed, and consequently the energy balance was improved compared to steam treatment.

Keywords: Sweet sorghum; Bagasse; Alkaline pretreatment; Microwave; Steam; Energy balance

Contact information: Banat's University of Agricultural Science and Veterinary Medicine "King Michael I of Romania", Timisoara, Calea Aradului Street, no. 119, 300645 Romania;
** Corresponding author: teo_vinti@yahoo.com*

INTRODUCTION

Current legislation requires European Union member states to ensure that renewable energy accounts for at least 14% of energy consumption in transportation by 2030. First outlined by the European Commission at the end of 2016 and officially adopted by the Council of the European Union at the end of 2018, the Renewable Energy Directive (REDII) sets a cap on first-generation biofuels made from food-based crops. To reduce greenhouse gas emissions from the increasing use of farm land for biofuel crops, this new law states that first-generation biofuels (from crops grown on agricultural land) should account for no more than 7% of energy consumption in transportation by 2020. The law contains milestones to boost the use of advanced biofuels to at least 0.2% of transportation energy in 2022, 1% in 2025, and 3.5% in 2030 (Biofuels International 2018; Pelkmans 2018).

Sweet sorghum is a versatile plant that can be used as a feedstock for both first- and second-generation bioethanol. Sweet sorghum juice is extracted from stalks and contains a mixture of sucrose, fructose, and glucose in various proportions and concentrations depending on several factors, such as the genetic variety, cropping technology, climate, and soil. Sweet sorghum juice can be concentrated to obtain a syrup or can be fermented to produce ethanol or other biochemicals (butanol, lactic acid, *etc*.). Current research has suggested that intensive plant breeding and harvesting methods could increase the sweet sorghum juice content to obtain ethanol yields of over 6000 L/ha (Dweikat 2008; Jia *et al*. 2013).

Unlike sweet sorghum juice, the lignocellulosic fraction from juice pressing, known as bagasse, has only non-food applications and is primarily used as an energy source. While an energy-intensive approach, such as fast pyrolysis of the lignocellulose, can produce syngas, bio-oil, and charcoal, this study proposed focusing on a biochemical approach driven by the new European legislation, namely the conversion of lignocellulose to second-generation biofuels, as an alternative to first-generation biofuels, which have a capped growth. In lignocellulosic bioethanol technology, pretreatment of biomass is required before hydrolysis and production of fermentable sugars. Lignocellulosic biomass is a complex structure, and it is recalcitrant to enzymatic hydrolysis during saccharification step to provide sugar monomers for fermenting microorganisms. Pretreatments are applied to break the complex cell wall structure, making more accessible the cellulose and hemicellulose from the complex to hydrolytic enzymes. The pretreatment step is the main impediment in the conversion of lignocellulose to biofuels and other biochemicals. Sweet sorghum bagasse (SSB) is no exception in this regard. Several research reports have studied different techniques for the pretreatment of SSB, including ammonia fiber expansion (Li *et al*. 2010), steam-SO_2 (Sipos *et al*. 2009), thermo-chemical pulping (Gonzalez *et al*. 2011), lime-microwave (Choudhary *et al*. 2012), alkaline (Xu *et al*. 2018), and hot water (Wang *et al*. 2012). As a general outcome from the previous research on the efficiency of pretreatment techniques for fermentable sugar production, the combination of chemical agents and thermal treatment has been reported to be more productive compared with single-agent pretreatments. For ethanol production, the accumulation of inhibitors, such as furfural and related compounds, leads to lower ethanol yields in the fermentation step. Previous research (Gonzalez *et al*. 2011; Jönsson and Martín 2016) studied pretreatment in acidic environments for releasing inhibiting compounds at higher concentrations compared with alkaline or thermal mediated pretreatments.

Regarding the energy source applied for thermal pretreatment, steam explosion is by far the most applied pretreatment technology for pilot scale applications in research and for industrial applications. The demonstration plant of Iogen (Canada) is running since 2004 and produces ethanol from wheat straw pretreated by a “modified” steam explosion. Other companies including Abengoa in Spain and USA, BetaRenewables in Italy, Clariant in Germany, or BP Biofuels in USA have applied steam treatments catalyzed or not catalyzed by chemicals (Harmsen *et al*. 2013). In contrast, microwave pretreatments are not applied in large scale biorefineries of lignocellulosic biomass, although laboratory research results indicate several advantages of microwave pretreatments comparing to conventional heating pretreatments. The most important advantage is that at the same temperature or even at lower temperatures and shorter reaction time, microwave pretreatments deliver higher saccharification ratios and enzymatic hydrolysis ratios compared to steam explosion (Li *et al*. 2016). Microwave radiation provides faster heating and can be integrated efficiently in continuous processes in contrast to steam treatments, where batch systems are widely applied because of the necessity for a confined vessel to reach high steam pressures. Microwave processing has already been applied in several fields, such as food processing, chemical engineering, and waste incineration. Microwave energy affects polar bonds, which causes disruptions that initiate chemical reactions, heating, and physical and biological modifications (Sridar 1998). In the case of biomass, microwaves cause heating, and chemical and mechanical changes to the lignocellulosic complex structure. These findings have been reported in previous studies (Azuma *et al*. 1984; Hu and Wen 2008; Saha *et al*. 2008; Zhao *et al*. 2010, Diaz *et al*. 2015), where microwave pretreatments were applied to assist the release of reducing sugars from various lignocellulosic biomass, including straw, corn stover, and perennial grass. Recently, there have been several studies regarding the microwave pretreatment of SSB (Choudhary *et al*. 2012; Chen *et al.* 2012; Marx *et al.* 2014), where lime, ammonia or sulphuric acid were used as a chemical agents.

After evaluation of the above-mentioned published studies on thermal-chemical pretreatments, sodium hydroxide (NaOH) was chosen as a preferred chemical agent with several considerations, such as a low production of inhibitors, high efficiency in de-structuring lignin, availability, facile neutralization by acids, *etc*. There is an important gap in establishing the optimum parameters in the pretreatment of SSB for the power, duration, and type of thermal energy in regards to the energy balance. It is important to evaluate the amount of energy consumed during pretreatment to the energy stored in the sugars released after hydrolysis of the pretreated bagasse. From this point of view, the optimum combination of wattage and exposure time in microwave pretreatment connected to sugars released in the hydrolysis phase needs to be determined.

This study compared the energy consumption and sugar yields obtained by applying microwave and steam thermal pretreatments. Consequently, the objectives of this study were to evaluate the effect of microwave pretreatment compared with steam pretreatment on the hydrolysis of SSB and sugar release in the presence of NaOH, and establish the optimum parameters for efficient energy consumption and sugar production from both the microwave and steam pretreatments.

EXPERIMENTAL

Materials

Biomass from *Sorghum bicolor* x *sudanese* hybrid Sugargraze was used as the biological raw material (seeds provided by Alta seeds, Irving, TX, USA, and cultivated in Copsa Mica, Romania). The stalks were harvested, the leaves and panicles were separated, and the juice was extracted by pressing using a roller press (Model TT1500D Pro 4 Roller – Tubo Biz, Tarlac City, Philippines). The resulting bagasse was fragmented to a 10-mm theoretical length using a cutting mill with a 10-mm mesh (SM100, Retsch GmbH, Haan, Germany). After milling, the sorghum biomass was dried in a drying oven (D.H.G. 9140-A, UNIEQUIP GmbH, Planegg, Germany) to an approximately 7% water content. A compositional analysis of the SSB was performed according to Sluiter *et al*. (2008). Chemicals used in this experiment were provided by Merck Timisoara, an affiliate of Merck KGaA, Darmstadt, Germany.

Pretreatment

The dried bagasse was partitioned into 125-mL Erlenmeyer flasks so that there was 2 g of SSB dry matter in each flask. The biomass was covered with 1% (w/v) NaOH solution in sufficient volume to soak and cover the whole amount of biomass in the flask. The flasks, which contained suspensions of SSB in 1% (w/v) NaOH solution, were subjected to different pretreatments. All of the SSB pretreatments were performed in triplicate.

Microwave pretreatment

The Erlenmeyer flasks containing SSB soaked in NaOH solution were placed in a 30-cm × 30-cm × 20-cm (L × W × H) domestic microwave oven (Samsung TDS, Samsung Electronics, Suwon, South Korea) with a total capacity of 2 kg per charge. Three microwave oven power settings were used, which were 150 W, 300 W, and 450 W. At each power setting, five treatment times were used, which were 5 min, 10 min, 15 min, 20 min, and 25 min. The main reason for choosing the microwave treatment conditions was to heat the biomass suspension to temperatures under 100 °C. When the temperature reach the boiling point of the liquid, any extra microwave energy goes to convert liquid into vapors and to replace lost heat. Replacing lost heat does not require as much energy as heating up. This mean that the energy used to generate microwaves when the temperature in the product raises over certain point, was wasted, so the efficiency of the pretreatment process decreased. By setting different values on the power change button in the microwave (150, 300, 450 W), the oven will turn on and off the magnetron, so that the issuance of microwaves and the exposure time changes. Intermittent function of the magnetron, changes the average power in order to get to the targeted total energy input and consequently the temperature of the sample in the oven also changes.

Steam pretreatment

The steam pretreatment was performed by placing three Erlenmeyer flasks containing SSB soaked in NaOH solution in a 13-L autoclave (Caloris R, Bucharest, Romania), with a total capacity of 3.25 kg of biomass per charge. The treatment time used was 10 min, and the pretreatment temperatures were 105 °C, 110 °C, 120 °C, 130 °C, and 140 °C.

After the pretreatments, all of the SSB suspensions were neutralized with 1% (w/v) H_2SO_4 solution until the pH was 5. To avoid pH variations, the SSB suspensions were stirred thoroughly using a magnetic stirrer for 30 min, and the pH was monitored. After pH correction, the SSB samples were washed five times with equivalent volumes of water to remove the pre-existing sorghum sugar that was not extracted by pressing. The sugars released during pretreatment and water-soluble inhibiting chemicals that could be formed during pretreatment were thus removed. The washing liquids were separated from the biomass *via* filtration using a vacuum pump (UNIJET, UNIEQUIP GmbH). All of the SSB samples were immediately hydrolyzed to avoid drying of the biomass. According to previous research (Brown and Torget 1996), lignocellulosic materials that have undergone an aqueous pretreatment must not be dried prior to hydrolysis because irreversible pore collapse can occur in the micro-structure of the biomass, which leads to a decreased enzymatic release of glucose from the cellulose. The risks are the same if the pretreated biomass is frozen for more than one month prior to hydrolysis (Vintila *et al*. 2010).

Enzymatic Hydrolysis

After the final wash, 50 mL of citrate buffer (pH = 5) were added to each pretreated sample (containing the equivalent of 2 g of dry matter). Commercial enzymatic product was added to the pretreated biomass. This study used the NS22192 cellulase complex, which is part of a Novozymes cellulosic ethanol enzyme kit (Novozymes, Bagsvaerd, Denmark). In this product, the key enzyme activity is provided by cellulase that hydrolyzes (1,4)-β-D-glycosidic linkages in cellulose and other β-D-glucans. The cellulolytic activity of the NS22192 cellulase complex was analyzed by a filter paper unit assay (Ghose 1987), and the amount of enzyme to be added to reach a minimum dose of 15 U of cellulase/g of cellulose was calculated.

According to the calculation, 75 µL of NS22192 cellulase complex were added to each flask containing the equivalent of 2 g of SSB dry matter. A final pH check was done before the samples were placed in a shaking incubator at 55 °C and 150 rpm. An enzymatic hydrolysis time of 24 h was set based on previous research using this enzymatic product (Vintila *et al*. 2016, 2017).

Analysis and Measurements

The electric power consumption of the microwave oven and autoclave was measured with a digital energy counter (GA Type EMT707CTL, EverFlourish Europe GmbH, Friedrichsthal, Germany). The energy consumed in each pretreatment was divided by the amount of biomass treated in the microwave oven or in the autoclave is referred as energy density (expressed in W/g).

Sugar yield refers to the amount in milligrams of reducing sugars released by hydrolysis of one gram of biomass (dry mater) after a pretreatment step with an energy input of one watt-hour. The measurement used for the initial biomass treatment and calculating the energy balance on this basis refers to and is applicable in laboratory equipments such as microwave oven and autoclaves, designed with this range of energy allowance. This study does not intend to transfer the obtained results directly to a larger scale. In microwave treatments, in laboratory scale ovens, the magnetron converts the high voltage to 2.45 GHz. Large industrial/commercial microwave equipments may use 915 megahertz magnetrons to excite the larger oven cavities. In this case, the study should be scaled up to that type of equipment.

After enzymatic hydrolysis, the hydrolysates were transferred to 50-mL centrifuge tubes and centrifuged at 10000 rpm for 30 min (Universal 320 R centrifuge; Hettich, Tuttlingen, Germany). The supernatant liquid was harvested for sugar analysis and the residual solid represented the unhydrolyzed biomass fraction. The centrifuge tubes containing the residual solid were dried at 105 °C. The weight of the dried biomass was calculated. The obtained value represented the unhydrolyzed fraction, and the biopolymer solubilisation was calculated knowing that each flask contained 2 g of SSB dry matter prior to pretreatment and hydrolysis.

The concentration of the reducing sugars released in the liquid phase during enzymatic hydrolysis was analyzed by the dinitrosalicylic acid method (Ghose 1987).

For statistical analysis, the experiments were performed with three replications to determine the optimum parameters of the SSB pretreatments with a full factorial design. Comparisons were made based on the standard errors and means.

RESULTS AND DISCUSSION

The chemical composition of sweet sorghum biomass varies depending on the geographic and climatic conditions, cultivation technology, and other factors. The SSB used in this experiment was composed of 31.27% (± 2.17%) cellulose, 22.24% (± 0.72%) hemicellulose, 17.43% (± 1.03%) insoluble lignin, 20.41% (± 0.82%) xylose, galactose, and mannose, and 1.80% (± 0.1%) arabinose. Additionally, the dried SSB contained 91.01% (± 0.43%) dry matter (90.85% (± 0.69%) organic matter) and 9.05% (± 0.66%) ash. The composition of the SSB compared well with other reported data, such as microwave-pretreated SSB (Choudhary *et al*. 2012) and sorghum bagasse (Sipos *et al*. 2009).

Effect of the Pretreatments on the SSB Biopolymer Solubilisation and Sugar Yield

The biopolymer solubilisation represents the percentage of total solids lost during the process of enzymatic hydrolysis. The biopolymer solubilisation denotes the portion of total insoluble biopolymers hydrolyzed into soluble components. This indicates the availability of cellulose from the lignocellulosic complex and the ability of the enzymes to break (1,4)-β-D-glycosidic linkages in the substrate. The high biopolymer solubilisation also reflects the decomposition degree of the lignocellulosic complex, presence of pores and cracks in the complex, and access of cellulases to the main substrates, cellulose, and hemicellulose.

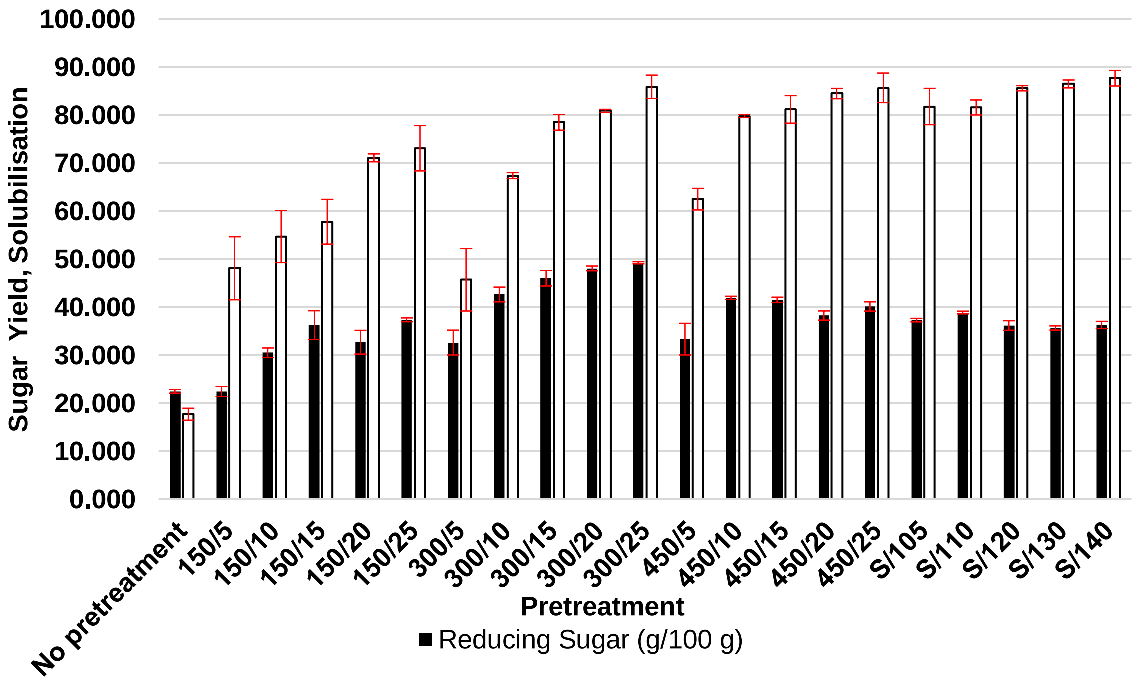

Fig. 1. Biopolymer solubilisation and reducing sugar yields of the SSB obtained after microwave and steam pretreatments with various conditions (power and durations in microwave and temperatures in steam treatments); on the X-axis, the labels with two values denotes microwave pretreatment for a certain power and duration, and S with a value denotes steam pretreatment at a certain temperature; for example, 150/5 is a microwave power of 150 W and an irradiation time of 5 min; and S/105 is steam pretreatment at 105 °C

Figure 1 shows that the biopolymer solubilisation (accessibility of enzymes to the substrate) was improved when a higher temperature and longer treatment time were applied. The first white bar on the left in Fig. 1 represents the biopolymer solubilisation of the unpretreated bagasse (17.7%). Heating the SSB suspension in NaOH solution in the microwave oven for 5 min at 150 W reached 93 to 95 ^{o}C, degraded the lignocellulosic complex, and mediated the access of the enzymes to the substrate, which enabled the hydrolysis of 50% of the glycosidic linkages in the cellulose and other β-D-glucans (second white bar in Fig. 1). A further increase in the reaction time and energy applied to the biomass led to higher biopolymer solubilisation rates. The last five white columns on the right side of Fig. 1 represent the biopolymer solubilisation of the steam-pretreated SSB. This type of pretreatment was more efficient in terms of the biopolymer solubilisation than the microwave pretreatment. However, the biopolymer solubilisation only partially reveals the efficiency of the pretreatments, as it does not show the portion of readily fermentable sugars released during hydrolysis. Therefore, the amount of reducing sugars released in the enzymatic hydrolysis phase was analyzed. The obtained values were represented in grams of reducing sugars to 100 g of SSB dry matter and are displayed in Fig. 1 as black bars. It was observed that the values correlated at low power settings (150 W to 300 W in the microwave treatments), where increased biopolymer solubilisation rates resulted in higher sugar yields. When high power settings were used in the biomass suspension (450 W in the microwave treatment) and an increased temperature in the autoclave from 105 °C to 140 °C, the reducing sugars concentration did not increase with an increase in the biopolymer solubilisation. Regarding values of temperature obtained in biomass suspensions during microwave treatments, the following indications were observed. In all runs, except irradiation for 5 minutes at 150 W, the boiling of biomass suspensions was observed. In the case of treatment for 5 minutes at 150 W, the temperature of biomass suspensions measured immediately after irradiation were in the range of 93 to 95 ^{o}C. In all other cases the temperature of boiling suspensions probably exceeded 100 ^{o}C. The temperature was not measured during microwave irradiation, because the temperature probe of the thermometer would be affected by microwaves. It should be mentioned that the glass flasks were not sealed and the vapors were all the time at atmospheric pressure. These results indicated that although the lignocellulosic complex was degraded efficiently at a high temperature, and the enzymes penetrated through the lignin polymer to reach the cellulosic substrate. The glucans formed were affected by the high temperature and a lower amount of sugar was released. This was attributed to sugar degradation and other reactions at temperatures above 140 ^{o}C, through processes known as Maillard reactions (Rizzi 2005). Rizzi (2005) indicated that sugars react with amine molecules from amino acids at high temperatures to form imine and amino ketone, which react further to form melanoidins under alkaline conditions. These compounds were reported to inhibit some bacterial species from the genres *Helicobacter*, *Escherichia*, *Bacillus*, *Salmonella*, *Pseudomonas*, *etc*. (Rufián-Henares and de la Cueva 2009), to be beneficial for *lactobacilli* and *bifidobacteria* (Ames *et al*. 1999), and no effect was reported for *Saccharomyces cerevisiae* (ethanol yeast). In fact, sugar molasses, which is rich in melanoidins, is used extensively in fermentation media to produce ethanol and yeast biomass. Consequently, a high temperature leads to sugar loss and the accumulation of by-products, such as melanoidins, which do not affect the fermenting microorganisms, but indirectly generate lower biomass-to-biochemical conversion rates.

Energy Balance in the Microwave and Steam Pretreatments of the SSB

Another important aspect of a pretreatment is the energy balance. It is important to know the amount of energy consumed during pretreatment to obtain a desired increase in the sugar yield during the hydrolysis phase. Therefore, this study compared the amount of energy (in W·h/g biomass dry matter) consumed in each pretreatment setup with the amount of energy produced from the released sugars during the hydrolysis phase. It was considered that 1 g of glucose can be theoretically converted to 0.51 g of ethanol (as per the stoichiometric reaction, 1 mol of glucose is converted by fermentation to 2 mol of ethanol and 2 mol of carbon dioxide). The average glucose to ethanol fermentation efficiency is 0.75 (in a pessimistic scenario). The theoretical yield of ethanol that is obtainable in practice from 1.0 g of glucose is 0.38 g. The energy density of ethanol is 25 MJ/kg (6.94 W·h/g) (Thomas 2000; Badger 2002; Heselton 2004). This energy density can be related to the glucose from which the ethanol was obtained by fermentation. Thus, 1 g of glucose can yield 0.38 g of ethanol by fermentation, which generates 2.64 W·h of energy. Hence, any pretreatment should be calibrated and reported with an energy balance using this value as the energy obtainable from 1 g of released glucose. This border line can be regarded as 0% energy efficient in terms of the pretreatment input/ethanol yield energy balance. This border line refers exclusively to the energy consumed during pretreatment. Any other energy inputs in the entire process of ethanol production were not quantified.

Table 1. Energy Consumption (Energy Density), Sugar Released, and Sugar Yields in the Microwave and Steam Alkaline Pretreatments of the SSB

Treatment	Time (min) in Microwave / Steam Temperature (°C)	Energy Density (W/g biomass dry matter)	Reducing Sugars Released (mg/g)		Calculated Sugar Yield (mg/g/W·h)	Efficiency*, (W·h/g)
			Mean	Standard deviation		
No treatment	0	0	224.7	3.81	-	0
Microwave (150 W)	5	2.1	224.5	10.46	106.9	0.0094
	10	4.2	307.0	9.99	73.1	0.0137
	15	6.3	362.6	29.92	57.6	0.0174
	20	8.3	365.0	24.74	44.0	0.0227
	25	10.4	373.1	3.97	35.9	0.0279
Microwave (300 W)	5	4.2	326.3	25.88	77.7	0.0129
	10	8.3	426.4	15.41	51.4	0.0195
	15	12.5	460.2	16.04	36.8	0.0272
	20	16.5	480.4	4.96	29.1	0.0343
	25	20.8	492.5	2.20	23.7	0.0422
Microwave (450 W)	5	6.3	333.4	33.02	52.9	0.0189
	10	12.5	419.8	3.31	33.6	0.0298
	15	18.8	415.4	5.46	22.1	0.0453
	20	25	410.6	9.50	16.4	0.0609
	25	31.3	401.4	9.61	12.8	0.0780
Steam	105	6.0	458.6	3.70	76.4	0.0131
	110	6.7	388.9	2.88	58.0	0.0172
	120	7.3	362.4	9.88	49.6	0.0201
	130	8.0	362.6	4.34	45.3	0.0221
	140	8.7	362.9	7.62	41.7	0.0240

** Energy consumed to release 1 g of reducing sugar*

In this study, the lowest efficiency in terms of energy quantity consumed to release 1 g of reducing sugar was achieved with microwave pretreatment at 450 W for 25 min, which released 12.8 mg of sugar from 1.0 g of biomass per 1.0 W·h (Table 1). This meant that 0.078 W·h of energy was consumed to release 1.0 g of sugar, which was 1/33 of 2.64 W·h. One of the highest efficiencies in terms of the energy quantity consumed and sugar yield was achieved with steam pretreatment at 105 °C, which released 76.4 mg of sugar from 1.0 g of biomass per 1.0 W·h. This meant that 0.013 W·h of energy was consumed to release 1.0 g of sugar, which was 1/200 of the energy that can be produced by fermenting 1 g of glucose to ethanol. When higher amount of energy is used to reach higher temperatures in autoclave, the amount of sugars released in the hydrolysis phase did not increased. This led to increase of energy density and decrease of sugar yield reported to the energy consumed to achieve higher steam pressures.

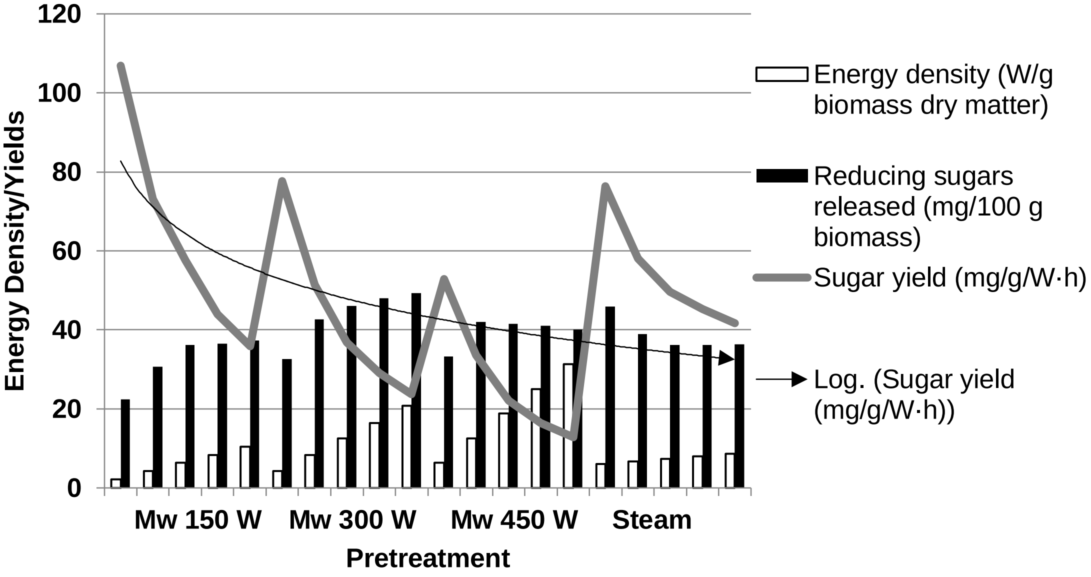

Fig. 2. Comparison of the energy consumption and sugar yields from the microwave and steam alkaline pretreatments of the SSB (Mw = microwave)

If for certain reasons (equipment costs, cost-benefit analysis, availability of mature technology at certain scale, other economic or technological reasons), an optimum microwave treatment should be recommended over steam treatment, then the application of microwaves for 10 min with a power of 300 W is recommended (Fig. 2). This setup generated high sugar yields from the biomass (426.4±15.41 mg of reducing sugars / 1 g of biomass dry matter) and medium sugar yields for the energy consumed (51 mg/g/W·h). This meant that 0.019 W·h of energy was consumed to release 1.0 g of sugar, which was 1/138 of 2.64 W·h.

These findings regarding sugar yields from the microwave pretreatment of lignocellulose were in accordance with other reported results (Ethaib *et al.* 2015), which indicated high sugar yields at a low microwave power (100 W to 150 W), a slight increase in the sugar yields up to 300 W, and a decrease in the sugar yields when the power was increased to 450 W. Although other research results indicated an increase in the sugar yields after alkali-microwave assisted treatment of lignocellulose when the microwave power was high (650 W to 800 W) (Ethaib *et al.* 2015), this study demonstrated that the increase in the sugar yields (at least in case of lignocellulose in SSB) is inefficient compared with the energy consumed at high microwave powers.

Although it has its own advantages such as non-thermal effect, the main disadvantage of this pretreatment was regarded to be the cost, as the heating generation by electricity (microwave are generated by electricity) is much more costly than other ways to generate heat. From this point of view, the higher efficiency of microwave pretreatments can be offset by low cost techniques to generate heat.

CONCLUSIONS

1. Pretreatment was essential to producing fermentable sugars from the sweet sorghum bagasse (SSB). In this study, a NaOH solution was used as a co-factor to break down the lignocellulosic complex in association with microwave and steam pretreatments.
2. In the steam pretreatment, it was not necessary to heat the sorghum bagasse above 105 °C because the sugar yields decreased with a steam temperature above this value.
3. In the microwave pretreatment, an application of microwaves for 10 min with a power of 300 W is recommended. With these parameters, some increase in the sugar yields was obtained, but the efficiency of the process decreased when the energy balance was calculated.
4. The energy balance was calculated as the proportion of energy consumed in the pretreatments to the energy that can be produced by fermenting 1 g of glucose to ethanol. In this study, the energy consumed in the different pretreatments ranged from 1/33 to 1/200 of the energy obtainable by fermenting released glucose to ethanol.
5. Overall, SSB pretreatment to produce fermentable sugars was effective. The energy balance was attractive in both the microwave and steam treatments and may be feasible for scaling up in the sweet sorghum biorefinery industry.

ACKNOWLEDGMENTS

The authors would like to thank the Institute of Applied Biotechnology and BUASVM "*King Michael I of Romania*" Timisoara for the financial support.

REFERENCES CITED

Ames, J. M., Wynne, A., Hofmann, A., Plos, S., and Gibson, G. R. (1999). "The effect of a model melanoidin mixture on faecal bacterial populations *in vitro*," *Brit. J. Nutr.* 82(6), 489-495.

Azuma, J.-I., Tanaka, F., and Koshijima, T. (1984). "Enhancement of enzymatic susceptibility of lignocellulosic wastes by microwave irradiation," *J. Ferment. Technol.* 62(4), 337-384.

Badger, P. C. (2002). “Ethanol from cellulose: A general review,” in: *Trends in New Crops and New Uses*, J. Janick, and A. Whipkey (eds.), ASHS Press, Alexandria, VA, pp. 17–21.

Biofuels International (2018). “UPM and LSB speak out over finalized REDII policy,” (https://biofuels-news.com/display_news/14186/upm_and_lsb_speak_out_over_finalised_redll_policy), Accessed on 15th Dec 2018.

Brown, L., and Torget, R. (1996). *Enzymatic Saccharification of Lignocellulosic Biomass* (LAP-009), NREL Ethanol Project, Golden, CO.

Chen, C., Boldor, D., Aita, G., and Walker, M. (2012). “Ethanol production from sorghum by a microwave-assisted dilute ammonia pretreatment,” *Bioresour. Technol.* 110, 190-197. DOI: 10.1016/j.biortech.2012.01.021

Choudhary, R., Umagiliyage, A. L., Liang, Y., Siddaramu, T., Haddock, J., and Markevicius, G. (2012). “Microwave pretreatment for enzymatic saccharification of sweet sorghum bagasse,” *Biomass Bioenerg.* 39, 218-226. DOI: 10.1016/j.biombioe.2012.01.006

Diaz, A.B., Souza Moretti, M.M., Bezerra-Bussoli, C., Costa Carreira Nunes, C., Blandino, A., da Silva, R., and Gomes, E. (2015). “Evaluation of microwave-assisted pretreatment of lignocellulosic biomass immersed in alkaline glycerol for fermentable sugars production,” *Bioresour. Technol.* 185, 316-323. DOI: 10.1016/j.biortech.2015.02.112

Dweikat, I. (2008). “Sweet sorghum research,” *University of Nebraska-Lincoln*, (http://agronomy.unl.edu/sweetsorghum), Accessed on 15th Dec 2018.

Ethaib, S., Omar, R., Kamal, S. M. M., and Biak, D. R. A. (2015). “Microwave-assisted pretreatment of lignocellulosic biomass: A review,” *Journal of Engineering Science and Technology* 10, 97-109.

Ghose, T. K. (1987). “Measurement of cellulase activities,” *Pure Appl. Chem.* 59(2), 257-268. DOI: 10.1351/pac198759020257

Gonzalez, R., Jameel, H., Chang, H.-M., Treasure, T., Pirraglia, A., and Saloni, D. (2011). “Thermo-mechanical pulping as a pretreatment for agricultural biomass for biochemical conversion,” *BioResources* 6(2), 1599-1614. DOI: 10.15376/biores.6.2.1599-1614

Harmsen, P., Lips, S., and Bakker, R. (2013). ”Steam explosion,” in: *Pretreatment of Lignocellulose for Biotechnological Production of Lactic Acid; Research Review,* Wageningen UR Food and Biobased Research, Wageningen, Netherlands, pp. 25.

Heselton, K. E. (2004). *Boiler Operator’s Handbook*, Fairmont Press, Lilburn, GA.

Hu, Z., and Wen, Z. (2008). “Enhancing enzymatic digestibility of switchgrass by microwave-assisted alkali pretreatment,” *Biochem. Eng. J.* 38(3), 369-378. DOI: 10.1016/j.bej.2007.08.001

Jia, F., Chawhuaymak, J., Riley, M. R., Zimmt, W., and Ogden, K. L. (2013). “Efficient extraction method to collect sugar from sweet sorghum,” *J. Biol. Eng.* 7(1), 1-8. DOI: 10.1186/1754-1611-7-1

Jönsson, L. J., and Martín, C. (2016). “Pretreatment of lignocellulose: Formation of inhibitory by-products and strategies for minimizing their effects,” *Bioresour. Technol.* 199, 103-112. DOI: 10.1016/j.biortech.2015.10.009

Li, B.-Z., Balan, V., Yuan, Y.-J., and Dale, B. E. (2010). “Process optimization to convert forage and sweet sorghum bagasse to ethanol based on ammonia fiber expansion (AFEX) pretreatment,” *Bioresour. Technol.* 101(4), 1285-1292. DOI: 10.1016/j.biortech.2009.09.044

Li, H., Qu, Y., Yang, Y., Chang, S., and Xu, J. (2016). ”Microwave irradiation – A green efficient way to pretreat biomass,” *Bioresour. Technol.* 199, 34-41. DOI: 10.1016/j.biortech.2015.08.099

Marx, S., Ndaba, B., Chiyanzu, I., and Schabort, C. (2014). “Fuel ethanol production from sweet sorghum bagasse using microwave irradiation,” *Biomass and Bioenergy* 65, 145-150. DOI: 10.1016/j.biombioe.2013.11.019

Pelkmans, L. (2018). *IEA Bioenergy Countries' Report – Update 2018: Bioenergy Policies and Status of Implementation*, IEA Bioenergy, Golden, CO.

Rizzi, G. P. (2005). “The Maillard reaction in foods,” in: *Maillard Reactions in Chemistry, Food, and Health*, T. P. Labuza, V. M. Reineccius, V. M. Monnier, J. O'Brien, and J. W. Baynes (eds.), Woodhead Publishing Ltd., Sawston, UK, pp. 11-19.

Rufián-Henares, J. A., and de la Cueva, S. P. (2009). “Antimicrobial activity of coffee melanoidins–A study of their metal-chelating properties,” *J. Agr. Food Chem.* 57(2), 432–438. DOI: 10.1021/jf8027842

Saha, B. C., Biswas, A., and Cotta, M. A. (2008). “Microwave pretreatment, enzymatic saccharification and fermentation of wheat straw to ethanol,” *J. Biobased Mater. Bio.* 2(3), 210-217. DOI: 10.1166/jbmb.2008.412

Sipos, B., Réczey, J., Somorai, Z., Kádár, Z., Dienes, D., and Réczey, K. (2009). “Sweet sorghum as feedstock for ethanol production: Enzymatic hydrolysis of steam-pretreated bagasse,” *Appl. Biochem. Biotech.* 153(1-3), 151-162. DOI: 10.1007/s12010-008-8423-9

Sluiter, A., Hames, B., Ruiz, R., Scarlata, C., Sluiter, J., Templeton, D., and Crocker, D. (2008). *Determination of Structural Carbohydrates and Lignin in Biomass* (NREL/TP-510-42618), National Renewable Energy Laboratory, Golden, CO.

Sridar, V. (1998). “Microwave radiation as a catalyst for chemical reactions,” *Curr. Sci. India* 74(5), 446-450.

Thomas, G. (2000). “Overview of storage development DOE hydrogen program,” in: *US DOE Hydrogen Program 2000 Annual Review*, San Ramon, CA.

Vintila, T., Dragomirescu, M., Croitoriu, V., Vintila, C., Barbu, H., and Sand, C. (2010). “Saccharification of lignocellulose - with reference to *Miscanthus* - using different cellulases,” *Rom. Biotech. Lett.* 15(4), 5498-5504.

Vintila, T., Gherman, V. D., Popa, N., Popescu, D., Buzatu, C., and Motoc, M. (2017). “Influence of enzymatic cocktails on conversion of agricultural lignocellulose to fermentable sugars,” *Rev. Chim.-Bucharest* 68(2), 373-377.

Vintila, T., Negrea, A., Barbu, H., Sumalan, R., and Kovacs, K. (2016). “Metal distribution in the process of lignocellulosic ethanol production from heavy metal contaminated sorghum biomass,” *J. Chem. Technol. Biot.* 91(6), 1607-1614. DOI: 10.1002/jctb.4902

Wang, W., Zhuang, X., Yuan, Z., Yu, Q., Qi, W., Wang, Q., and Tan, X. (2012). “Effect of structural changes on enzymatic hydrolysis of eucalyptus, sorghum bagasse, and sugarcane bagasse after liquid hot water pretreatment,” *BioResources* 7(2), 2469-2482. DOI: 10.15376/biores.7.2.2469-2482

Xu, J., Li, M., and Sun, R. (2018). “Successive fractionations of hemicelluloses and lignin from sorghum stem by sodium hydroxide aqueous solutions with increased concentrations,” *BioResources* 13(2), 2356-2373. DOI: 10.15376/biores.13.2.2356-2373

Zhao, X., Zhou, Y., Zheng, G., and Liu, D. (2010). "Microwave pretreatment of substrates for cellulase production by solid-state fermentation," *Appl. Biochem. Biotech.* 160, 1557-1571. DOI: 10.1007/s12010-009-8640-x

Article submitted: January 10, 2019; Peer review completed: March 9, 2019; Revised version received: March 20, 2019; Accepted: March 25, 2019: Published: March 29, 2019.
DOI: 10.15376/biores.14.2.4022-4034