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Comparison of steam-assisted versus microwave-assisted treatments for the fractionation of sweet sorghum bagasse

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Abstract

Background: The fractionation of lignocellulosic materials can be applied to get each of the components (cellulose, hemicellulose and lignin) of biomass in its maximum purity and yield. The individual component can be further processed to high-value products such as fuels and biomaterials at existing or newly developing biorefineries. The steam-assisted and microwave (MW)-assisted processes were used to fractionate sweet sorghum bagasse into hemicellulosic sugars, cellulose-rich and high-density residue and solid lignin. The treatment temperature evaluated for the fractionation process was 121 °C for 30 to 120 min. The substrate was autohydrolysed to extract the hemicellulose, and the residue was delignified using lime solution to obtain cellulose-rich residue. The lignin and lime from the liquor obtained after the lime treatment of the autohydrolysed sweet sorghum bagasse (SSB) was precipitated using carbon dioxide gas.

Results: Under optimum conditions, the steam-assisted autohydrolysis extracted 72.69 (±0.08) % by weight of the hemicelluloses while the MW-assisted autohydrolysis extracted 70.83 (±0.49) % of the hemicelluloses from the sweet sorghum bagasse. The steam-assisted lime treatment resulted in 69.67 (±1.26) % of the lignin extraction the MW-assisted lime treatment resulted in 68.27 (±1.19) % of the lignin extraction from the corresponding autohydrolysed sweet sorghum bagasse samples. The CO_2 treatment precipitated 58.85 (±3.2) % of the lignin dissolved in the yellow liquor of the steam-assisted process while 60.26 (±2.11) % of the dissolved lignin was recovered from the yellow liquor of the MW-assisted process.

Conclusions: The two methods have not exhibited significant differences in overall recovery of the solids, hemicellulose extraction, delignification, residual concentration of cellulose and ash or in the recovery of lignin and lime. The difference was significant (*p* value <0.05) only in the concentration of total reducing sugars in the hydrolysate and the yellow liquor. The MW-assisted process increased the total crystallinity index (TCI) of the cellulose in the treated SSB and also increased the concentration of guaiacyl lignin content in the recovered lignin which was thermally more stable than the lignin produced in the steam-assisted process.

Keywords: Sweet sorghum bagasse; Steam-assisted treatment; Microwave-assisted treatment; Hemicellulose extraction; Delignification; Recovery of lignin

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Background

Innovative research on the fractionation of lignocellulosic biomass is essential for the sustainable production of intermediary materials and bioproducts for the direct industrial applications. Multi-step processes producing multi-products need to be established for the effective conversion of lignocellulosic materials in large-scale biorefineries [1]. The fractionation of lignocellulosic materials is applied to get each of the components (cellulose, hemicellulose and lignin) of biomass in its maximum purity and yield. The individual component can be further processed to high-value products such as fuels and biomaterials at existing or newly developing biorefineries [1, 2].

Fractionation of lignocellulosic materials can be achieved with the use of chemicals such as acids, alkalis, water and ionic liquids [1]. Hydrothermal fractionation and obtaining the hemicellulosic products from lignocellulosic biomass is considered as the first process in the biorefinery operations. Hydrothermal treatment of biomass is highly dependent on the behaviour of water at subcritical conditions. The ionization constant of water increases with increase in temperature and provides an acidic condition for the hydrolytic reactions in the medium. The decrease in dielectric constant of water with increase in temperature at subcritical conditions facilitates the dissolution of organic compounds from biomass [3]. The increase in the severity in treatment conditions would lead to increase in degradation of hemicellulosic sugars. Studies have shown that lower reaction temperatures for longer residence times favour the pentose sugar recovery [3].

Extraction of lignin from the hemicellulose extracted (autohydrolysed) lignocellulosic materials can be achieved through the use of alkaline materials like calcium hydroxide (lime). The effectiveness of the delignification using lime would be higher in autohydrolysed lignocellulosic materials than in the untreated biomass [4, 5]. Extraction of lignin from the autohydrolysed lignocellulosic materials can be applied to produce a solid residue rich in cellulose and with higher bulk density than untreated and autohydrolysed biomass. Moreover, the use of lime for the extraction of lignin from the autohydrolysed biomass has the advantage of recovering the lime and lignin using carbon dioxide gas [4]. Thus, hydrothermal treatment followed by lime treatment can be applied for the industrial fractionation of lignocellulosic materials into multiple products in an economic and eco-friendly way [6].

The mode of energy supply for the fractionation of lignocellulosic biomass is important, and it may also affect the yield and overall efficiency of the fractionation process. Generally, steam is used to supply energy for myriad of industrial processes [7]. However, microwave heating is an efficient method of energy supply for the treatment of biomass for the production of high-value products from it [8, 9]. Microwave-assisted pretreatment processes are based on the selective heating of the lossy components in the lignocellulosic materials that leads to the disruption in the original structure of lignocelluloses. Cellulose component has low loss tangent factor (tan δ) and generate less heat whereas the lignin and water have high loss tangent factor (tan δ) and can generate high heat when the lignocelluloses are treated with microwaves [10].

The effect of microwave on the depolymerization of the lignocellulosic components is attained through the reduction in glass transition temperature. The added water for the treatment of biomass reduces the glass transition temperature of polymers in biomass and thus facilitates the interaction of the individual molecules with the microwaves to generate heat. The lignocelluloses heated in an aqueous medium releases acetic acid that leads to the hydrothermal treatment of the substrate [10]. Presence of ionic compounds like calcium hydroxide further increases the loss tangent factor (tan δ), which measures the heating ability of the substance under microwave and of the aqueous solutions, and facilitates the reactions in biomass mixture [11]. Moreover, microwave heating can reduce the reaction times from hours to minutes with minimum side reactions and improve yields and reproducibility of the reactions [12].

In this work, a comparison was made between the outcomes of the fractionation of sweet sorghum bagasse (SSB) in an autoclave and in a microwave apparatus. The SSB was fractionated into hemicellulosic sugars, cellulose and lignin at 121 (±0.5) °C in both cases. The isothermal treatment time was 90 min for the extraction of hemicellulose through hydrothermal treatment and 106 min for the extraction of lignin from the autohydrolysed residue. The comparison was made on the efficiency of each process on the extraction of hemicellulose as reducing sugars, extraction of lignin from the autohydrolysed biomass, recovery and purity of cellulose and lignin, and the physicochemical properties of the products produced through the two processes.

Methods

Substrates and the treatments

SSB powder was used as the substrate for the steam-assisted hydrothermal treatment (SAHT) and microwave-assisted hydrothermal treatment (MAHT) studies. Sweet sorghum (*Sorghum bicolor* L. Moench, CSSH-45) was collected from the experimental field (45° 26' 01" N, 73° 55' 53" W) of the Department of Plant Science of McGill University. The harvested plants were defoliated, and the sweet juice was extracted by crushing the stems. The free sugars present were removed by washing the bagasse using cold water [13]. The bagasse obtained was then dried in a hot air oven at 50 °C for 72 h to obtain constant dry mass. The dried bagasse was chopped to 1–2-cm-long pieces using scissors and pulverized using a blender-grinder (Homeland Housewares, LA, USA). The powder was stored in plastic air-tight containers and was used after drying at 105 °C for 24 h for treatments and analyses [14, 15].

The SSB powder was initially subjected to the hydrothermal treatments at 121 (±0.5) °C for 30 to 120 min with substrate concentration varying from 10 to 20 % by weight of the mixture. The optimum conditions to get maximum hemicellulose extraction and solids recovery were found to be 13 % substrate concentration and 90 min of isothermal treatment [14]. The residue obtained from the SAHT was dried and then subjected to the steam-assisted lime treatment (SALT) at 121 (±0.5) °C for 30 to 120 min with 10 % of the mixture substrate concentration and calcium hydroxide dosage varying from 10 to 20 % by weight of the substrate. The optimum conditions for the maxima of lignin extraction and solids recovery were found to be 10 % of the substrate calcium dosage and 106 min of isothermal treatment residence time [16]. Similarly, the residue obtained from the MAHT was dried and then subjected to the MW-assisted lime treatment (MALT) at 121 (±0.5) °C for 30 to 120 min with 10 % by weight of the mixture substrate concentration and calcium hydroxide dosage varying from 10 to 20 % by weight of the substrate. The optimum conditions for the maxima of lignin extraction and solids recovery were found to be 10 % of the substrate calcium dosage and 78 min of isothermal treatment residence time.

The yellow-coloured liquor containing lignin and calcium hydroxide was obtained as the liquid product of the lime treatment. The yellow liquor was bubbled with carbon dioxide to recover calcium hydroxide, as calcium carbonate and the lignin are present in it [17]. The schematic of the treatment process is shown in Fig. 1.

Steam-assisted treatments were conducted in an autoclave (DSE-8000, NAPCO, USA), and the microwave-assisted treatments were conducted in a microwave apparatus (MiniWave, SCP Science, Canada).

Experimental designs

The effects of independent variables such as substrate concentration and isothermal treatment residence time on the hydrothermal treatment responses were investigated by using a response surface design prepared using the JMP® 11 software (SAS Institute USA). Similar experimental design was used for the lime treatment of the autohydrolysed SSB where instead of varying the substrate concentration, the calcium hydroxide dosage was varied. The response surface design (Table 1) was used to reduce the number of experimental runs as well as to obtain a response surface showing the individual, cumulative and interactive effects of the independent factors [18]. The responses were fitted to the second-order polynomial regression equations (Equation 1) to understand the effect of the isothermal treatment residence time and substrate concentration (in hydrothermal treatment) or the lime concentration (in lime treatment).

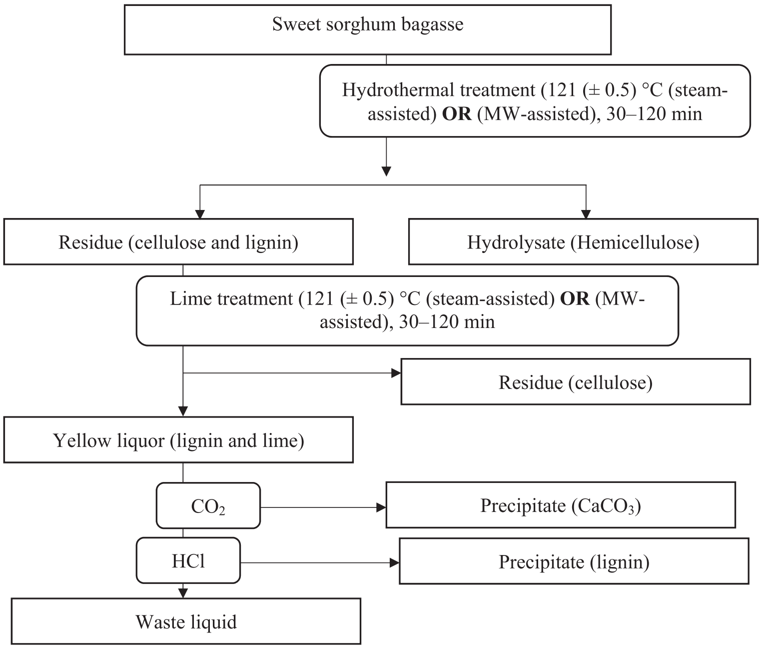

Fig. 1 The schematic of the process for the treatment of SSB using the steam-assisted or the MW-assisted method

Table 1 The response surface experimental design used in the studies

Run	Pattern	Substrate OR $Ca(OH)_2$ concentration (%)	Treatment residence time (min)
1	−−	10	30
2	−+	10	120
3	a0	10	75
4	0a	15	30
5	++	20	120
6	A0	20	75
7	0A	15	120
8	00	15	75
9	+−	20	30
10	00	15	75

$$Y = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_{12} x_1 x_2 + \beta_{11} x_1^2 + \beta_{22} x_2^2 \tag{1}$$

Where Y is the response such as hemicellulose hydrolysis, solid recovery, sugar degradation, lignin extraction, etc., x_1 and x_2 are the independent factors such as isothermal treatment residence time and substrate concentration or lime concentration, β_0 is the intercept, β_1 and β_2 are the linear coefficients, β_{12} is the interaction coefficient and β_{11} and β_{22} are the quadratic coefficients.

Each treatment including the two centre points (15 % substrate concentration (in hydrothermal treatment) or 15 % calcium hydroxide dose (in lime treatment) and 75 min of treatment time) was conducted in triplicates, and the solid residues obtained after the treatment were analysed in triplicates for cellulose, hemicellulose, lignin and mineral concentrations. Similarly, the liquid samples (hydrolysate) were analysed for hexose and pentose sugars, total carbohydrates, sugar degradation products (hydroxymethyl furfural (HMF) and furfural) and dissolved lignin. The process conditions were optimized to obtain the maximum hydrolysis of hemicellulose (in hydrothermal treatment) and maximum extraction of lignin (in lime treatment) and recovery of solids with minimum sugar degradation.

The carbon dioxide treatment for the yellow liquor was applied by following a completely randomized design of experiment with one factor (final pH of the yellow liquor) at multiple levels, from pH 2.5 to 12 with a pH value interval of 0.5 [17].

Analytical methods

The particle size distribution of the SSB powder was estimated using sieve analysis [19], and the bulk density of the solid samples produced through the steam-assisted and microwave-assisted methods was estimated using a 100-mL measuring cylinder [20]. The dielectric properties of the SSB slurries with water and lime solution at 2450 MHz were measured by using a network analyser equipped with a probe (Agilent 8722 ES s-parameter, Agilent USA). The measurements were controlled, and the results were obtained using the software (Agilent 85070D E01.02, Santa Clara, USA) integrated with the network analyser [21].

Gravimetric methods for the analysis of forage fibers were used to estimate the concentration of cellulose, hemicellulose, lignin and minerals in the solid SSB samples before and after the treatments [15]. UV-Vis spectroscopy was used for measuring the concentration of hexose and pentose sugars, sugar degradation products and lignin in the liquid samples. The para-bromoaniline method was used for the estimation of pentose sugars. Heating of the sample at 70 °C for 10 min with para-bromoaniline reagent converts pentose sugars in the sample into furfural which in turn formed a pink coloured complex with para-bromoaniline. The pentose sugar concentration was estimated by measuring the absorbance at a wavelength of 520 nm. Concentration of pentose sugars in the samples was estimated from a standard curve prepared using D-xylose sugar [22].

The concentration of reducing sugars were estimated by the 2,4,6-tripyridyl-s-triazine (TPTZ) method. It is based on the oxidation of sugars using $Fe^{3+}(K_3[Fe(CN)_6])$ in alkaline media which leads to the formation of Fe^{2+}. TPTZ reacts with Fe^{2+} to form a violet-coloured complex that has a maximum absorption at a wavelength of 595 nm [23]. The total carbohydrate present in liquid samples was estimated by using the anthrone reagent method. It uses sulphuric acid to dehydrate the sugars into furfurals which in turn condenses with the anthrone reagent to form a green-coloured complex showing maximum absorbance at a wavelength of 620 nm [24].

Ultraviolet (UV) spectroscopy was used for the estimation of furfural and hydroxyl-methyl furfural (HMF) in liquid samples [25]. Furfural shows specific absorption maximum at 277 nm, and HMF shows specific absorption maximum at 285 nm. Lignin has a characteristic absorption at 280 nm which may interfere with the estimation of HMF and furfural. Sodium boro-hydride (NaBH4) was used to reduce furfural and HMF so that their absorption maxima at 277 and 285 nm respectively would disappear. The difference in absorption before and after the reduction was used to estimate the concentration of furfural and HMF in the samples [26]. Similarly, lignin solubilized in the liquid was estimated by UV-spectroscopy by measuring the absorbance at 205 nm [27].

Complexometric titration with ethylene-diamine-tetra-acetic acid (EDTA) was used for the estimation of calcium concentration in solid and liquid samples [28, 29]. In this method, 500 mg of the oven dried solid sample was burned to ash in a muffle furnace (Barnstead Thermodyne 48000, Thermo Scientific, USA) at 600 °C for 4 h, and the ash was completely dissolved in 10 mL of 0.5 N HCl and then diluted to 50 mL with distilled water. The solution was kept at a room temperature for 5 min after adding 6 mL of 1 M NaOH to make the pH of the solution to 13. Two drops of Eriochrome Black T (EBT) (Sigma–Aldrich, Canada) indicator solution were added, and the solution was titrated with 0.1 M EDTA (Sigma–Aldrich, Canada) until the colour changed from blue to red. Calcium concentration was estimated from the titrated values which were taken from the average of three titrations of a sample.

The solid samples were analysed using a differential scanning calorimeter (DSC) (Q100 V9.8, TA Instrument, Waters LLC), and the data was analysed using Universal Analysis 2000, for thermal property changes. For the DSC analyses, oven-dried samples between 9 and 10 mg were weighed and sealed in aluminum hermetic pans. The heating range was from 20 to 500 °C with a ramp up temperature of 10 °C/min with a Nitrogen flow rate of 50 mL/min to purge the heating chamber. The DSC thermograms of the SSB treated with the steam-assisted and microwave-assisted methods were compared against each other. Similarly, the DSC thermograms of the lignins precipitated from the yellow liquor produced through the two treatment methods were compared against each other for thermal properties such as glass transition temperature, crystallization temperature, melting temperature and decomposition temperature.

The solid samples were also analysed for functional group changes using the Fourier transform infrared (FTIR) spectroscopy (Nicolet iS5, Thermo Scientific, USA). The spectra of the SSB samples produced through the steam-assisted and microwave-assisted methods were compared to each other. Similarly, the spectra of the lignins precipitated from the yellow liquor produced through the two treatment methods were also compared against each other.

The IR absorption peak characteristic of a biomass component at a particular wavenumber and the corresponding bonds with characteristic movement are given in Table 2. The ratio of peak areas at 1510 and 900 cm^{-1} gives the ratio of lignin and cellulose [30]. The ratio of absorbance at 1428–1430 cm^{-1} to the absorbance at 898–900 cm^{-1} was used to estimate the total crystallinity index (TCI) of the samples [31, 32].

The physical changes in the solid samples were also analysed using scanning electron microscopy (SEM) (TM3000, Hitachi, USA), and the surface changes in the SSB samples treated with steam-assisted and microwave-assisted methods were compared to each other. The oven-dried samples were kept under vacuum in the SEM microscope without any treatments or modifications.

Table 2 FTIR absorption peaks and characteristic bonds and biomass component [30, 53, 54]

Biomass component	Absorption peak	
Wavenumber (cm^{-1})	Characteristic bond and movement	
Hemicellulose	1737	C=O stretching
	1056	C–O stretch
	1235	C–O stretch
	1375	C–H deformation
Lignin	1636	C=O stretching
	1516	Phenyl ring stretching
	1218	C–O of guaiacyl ring
	1268	C–O of guaiacyl ring
	1315	C–O of syringyl ring
	1329	Guaiacyl and syringyl condensed
	1502–1600	Aromatic skeletal vibration
	1235	C–O stretch
Cellulose	1158	C–O–C stretching
	900–901	Amorphous cellulose
	1033	C–O stretching at C_6
	1061	C–C stretching
	1114	C–O stretching at C_3
	1056	C–O stretch
	1428	Crystalline cellulose

Analysis of variance (ANOVA) was conducted to determine the statistical significance (p value ≤0.05) of the responses and to estimate the fit of the polynomial prediction equation to the observed responses. The difference in the response by the SSB towards steam-assisted and microwave-assisted treatments were analysed using the Student's *t*-test [33].

Results and discussion

Physicochemical properties of the sweet sorghum bagasse used for the treatments

The composition of SSB powder used for the treatments was estimated on a mass percent basis to be 45.03 (±0.03) % cellulose, 30.17 (±2.06) % hemicellulose, 24.40 (±3.02) % lignin and 0.78 (±0.22) % ash. The particle size distribution of the samples used for the treatment was 75–1000 μm estimated through the sieve analysis [19]. The bulk density of the raw SSB powder was 85.79 (±5.73) (kg/m^3) estimated using a 100-mL measuring cylinder [20].

Dielectric properties of the mixtures used in the microwave-assisted treatments

The dielectric properties of water and aqueous mixture of SSB at different concentrations were studied at 30, 50 and 80 °C, and the dielectric loss (tan δ) of the samples is shown in Fig. 2. The loss tangent (tan δ) is the ratio of dielectric loss (ε”) and dielectric constant (ε’) of the SSB mixture with water decreased with increase in temperature. The decrease in tan δ decreases the heat generation in aqueous solutions with increase in temperature. In comparison to the water, the presence of ionic compounds in biomass increases the tan δ and increases the heat production under microwave treatment [11]. The change in dielectric properties of the biomass mixture might have affected the responses of the dielectric heating of the biomass mixture for its fractionation.

The tan δ was increased with increase in temperature when the lime solution was used to prepare the mixture. The increase in the tan δ value for the calcium hydroxide solutions can be attributed to the increase in ions from calcium hydroxide and the dissociation of calcium hydroxide molecules to ions with the increase in temperature [11]. The increase in loss factor increases the heat generation in lime solutions with increase in temperature. The increase in tan δ value with increase in temperature will help in reduced energy input for obtaining higher temperatures. However, the energy input required for the MW-assisted treatments of SSB was not considered in this study.

Comparison of solids recovery during steam-assisted and microwave-assisted processing of sweet sorghum bagasse

The residual SSB recovered after the SAHT was more than that from the MAHT (Fig. 3). The SAHT of SSB, at 10 to 20 % by weight of substrate concentration and 121 (±0.5) °C for 30 to 120 min of treatment retention time, resulted in about 75.32 (±1.51) % solids recovered after the treatment. Under optimum conditions (13 % of substrate concentration and 90 min of isothermal treatment at 121 (±0.5) °C), the SAHT resulted in 75.36 (±0.16) % of solids recovery. The MAHT of SSB at 10 to 15 % by weight of substrate concentration and 121 (±0.5) °C for 30 to 120 min of isothermal treatment retention time resulted in about 70.42 (±3.41) % solids recovered after the treatment. Under optimum conditions for MAHT (10 % of substrate concentration and 65 min of isothermal treatment at 121 (±0.5) °C), the solids recovery was 72.52 (±0.31) %. The MAHT resulted in higher dissolution of solids into the hydrolysates, and the difference in quantity of solids recovered after the two treatments was found to be significant (p value <0.05). The higher dissolution of solids during MAHT can be attributed to the higher energy absorbed by the mixture during the MAHT [34] than during the SAHT within the total duration of the treatment [35]. The higher dissolution of solids will be required for the higher extraction of components from the biomass.

The MALT resulted in higher dissolution of solids into the yellow liquor than during the SALT, but the difference in quantity of solids recovered after the two treatments was not found to be significant. The SALT of the SAHT-SSB at 121 (±0.5) °C for 30 to 120 min with 10 % substrate concentration and 10–20 % by weight of the substrate $Ca(OH)_2$ dosage resulted in 77.84 (±8.09) % solids recovery. Under optimum conditions, the SALT (10 % $Ca(OH)_2$ dosage and 106 min of isothermal treatment at 121 (±0.5) °C) of the SAHT-SSB resulted in 76.75 (±0.15) % solids recovery. The MALT of the MAHT-SSB at 121 (±0.5) °C for 30 to 120 min with 10 % (g/g mixture) substrate concentration and 10–20 % of $Ca(OH)_2$ dosage resulted in 75.12 (±7.86) % solids recovery. Under optimum conditions, the MALT of the MAHT-SSB (10 % $Ca(OH)_2$ dosage and 78 min of isothermal treatment at 121 (±0.5) °C) resulted in 71.76 (±1.28) % solids recovery. The increased dissolution of solids during the MALT can be attributed to the higher energy absorption during the MW-assisted treatment and the accompanied higher dissolution of biomass components into the yellow liquor.

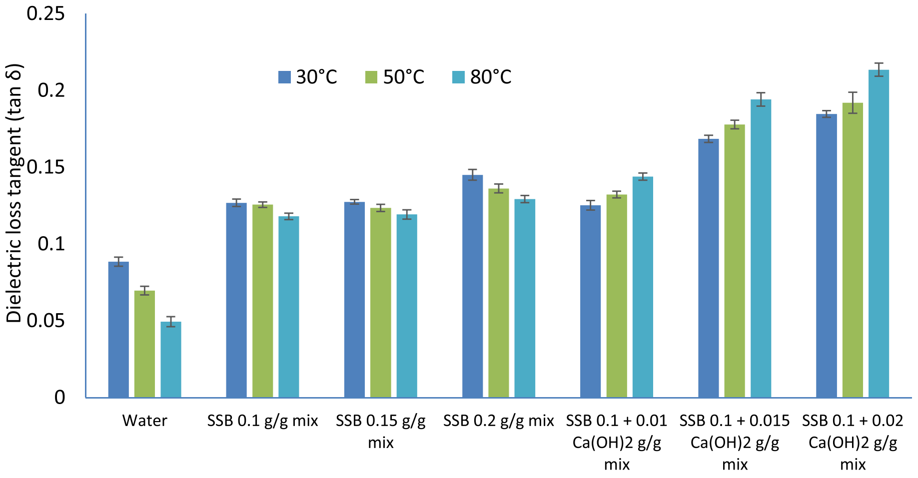

Fig. 2 The dielectric loss tangent of water and the SSB samples at different temperatures (the *error bars* are showing the ±standard deviation)

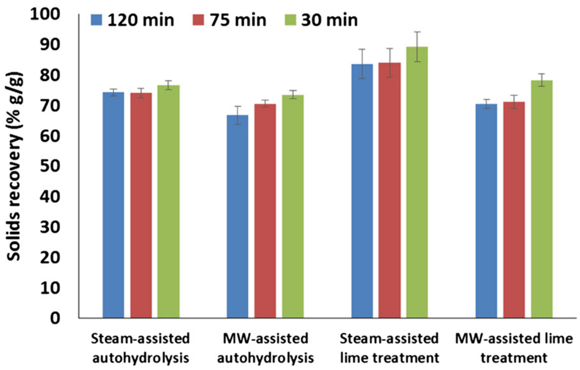

Fig. 3 Comparison of solids recovery during the steam-assisted and MW-assisted treatments of SSB (the *error bars* are showing the ±standard deviations)

Comparison of steam-assisted versus microwave-assisted treatments on hemicellulose extraction from sweet sorghum bagasse

The SAHT and MAHT have no significantly different effects on the extent of hemicellulose extraction from the SSB under the range of treatment conditions tested in this experiment. The quantities of hemicellulose extracted during the SAHT and the MAHT were 70.28 (±4.16) % and 69.92 (±7.63) %, respectively (Fig. 4). Under optimum conditions, the SAHT resulted in 72.69 (±0.08) % of hemicellulose hydrolysis. Similarly, under the optimum conditions for MAHT, the hemicellulose hydrolysis was 70.83 (±0.49) % from SSB. Therefore, in comparison to the steam-assisted fractionation, the MW-assisted fractionation process requires less duration of isothermal treatment to effect the similar amounts of hemicellulose extraction from SSB. However, there is no effect of microwaves on the extent of extraction of hemicellulose from SSB at 121 °C and the range of substrate concentration and treatment times investigated here.

The MALT resulted in higher extraction of hemicellulose into the yellow liquor than during the SALT of the autohydrolysed SSB. The average quantities of hemicellulose extracted during the SALT and the MALT were 19.79 (±7.31) % and 27.38 (±4.10) %, respectively. The difference in quantity of hemicellulose extracted between the two treatments was found to be significant (*p* value <0.05). Under optimum conditions, the SALT of the SAHT-SSB resulted in 24.19 (±1.06) % hemicellulose extraction, whereas under optimum conditions, the MALT of the MAHT-SSB resulted in 26.11 (±2.79) % hemicellulose extraction. The higher extraction of hemicellulose during MALT can be attributed to higher energy absorbed by the mixture and the resultant dissolution of the dry matter [34] within the total duration of the treatment [35].

Comparison of steam-assisted versus microwave-assisted treatments on lignin extraction from the sweet sorghum bagasse

Lignin is covalently bonded with hemicellulose, and during hydrothermal treatments, lignin-hemicellulose linkages undergo degradation leading to partial depolymerisation and relocation of the lignin component. It was observed that some amount of lignin was dissolved into the hydrolysate depending on the treatment conditions [3]. The SAHT and MAHT treatments have significantly different (*p* value <0.05) effects on the extent of lignin extraction from SSB. The average quantities of lignin extracted during the SAHT and the MAHT were 2.67 (±1.97) % and 4.47 (±1.17) %, respectively (Fig. 5). Under optimum conditions, the SAHT resulted in 1.46 (±0.04) % of lignin extraction into the hydrolysate, whereas under optimum conditions for MAHT, the lignin extraction was 3.80 (±0.17) % from SSB. The difference in the dissolution of lignin during the SAHT and MAHT of SSB samples may be attributed to the difference in the mode of energy supply in the two treatment methods. However, further studies are required to confirm the exact effect of microwaves on the breakage of the covalent bonds between lignin and hemicellulose components in biomass.

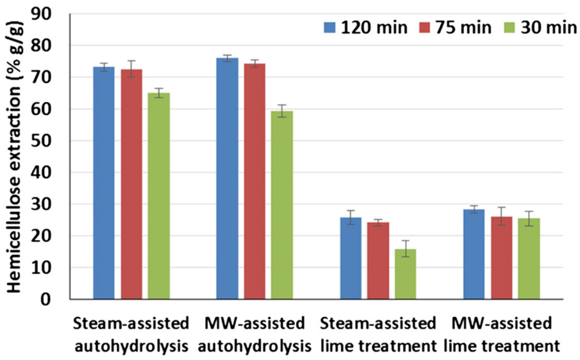

Fig. 4 Comparison of hemicellulose extraction during the steam-assisted and MW-assisted treatments of SSB (the *error bars* are showing the ±standard deviations)

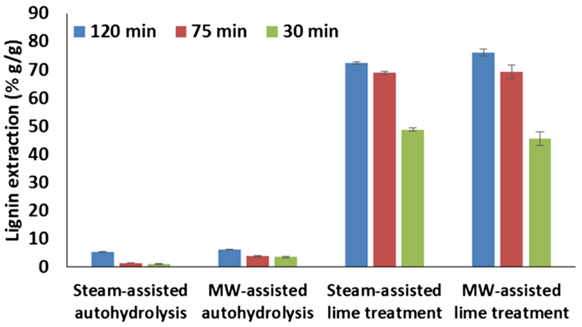

Fig. 5 Comparison of lignin extraction during the steam-assisted and MW-assisted treatments of SSB (the *error bars* are showing the ±standard deviations)

The MALT resulted in less extraction of lignin into the yellow liquor from MAHT-SSB than during the SALT of the SAHT-SSB. The quantities of lignin extracted during the SALT and the MALT were 67.66 (±9.89) % and 63.10 (±12.85) %, respectively. However, the difference in quantity of lignin extracted between the two treatments was found to be not significant. Under optimum conditions, the SALT of the SAHT-SSB resulted in 69.67 (±1.26) % lignin extraction, whereas under optimum conditions, the MALT of the MAHT-SSB resulted in 68.27 (±1.19) % lignin extraction. The study has shown that, in comparison to the steam-assisted fractionation, the microwave-assisted fractionation process require less duration of isothermal treatment to effect similar quantities of lignin extracted from SSB. Moreover, it was also shown that the microwave-assisted and steam-assisted methods have similar effect on the extent of extraction of lignin from the hemicellulose-extracted SSB within the range of treatment conditions investigated.

Comparison of steam-assisted versus microwave-assisted treatments on cellulose extraction from the sweet sorghum bagasse

In comparison to the hemicellulose, the cellulose and lignin components of lignocellulosic materials are less affected by the hydrothermal treatments at subcritical conditions [3]. The steam-assisted and microwave-assisted hydrothermal treatments have significantly different (p value <0.05) effects on the extent of cellulose hydrolysed from SSB. The quantities of cellulose hydrolysed during the SAHT and the MAHT were 5.53 (±0.98) % and 14.65 (±0.70) %, respectively (Fig. 6). Under optimum conditions, the SAHT resulted in 5.46 (±1.08) % of cellulose hydrolysed into the hydrolysate, whereas under optimum conditions for MAHT, the cellulose hydrolysis was 14.10 (±0.60) % from SSB. The increased dissolution of cellulose during MW-assisted treatment can be attributed to the increased breakage of bonds between cellulose and lignin and dissolution of them into the hydrolysates. The increased dissolution of cellulose can be the reason for the decreased solids recovery during the MW-assisted treatments.

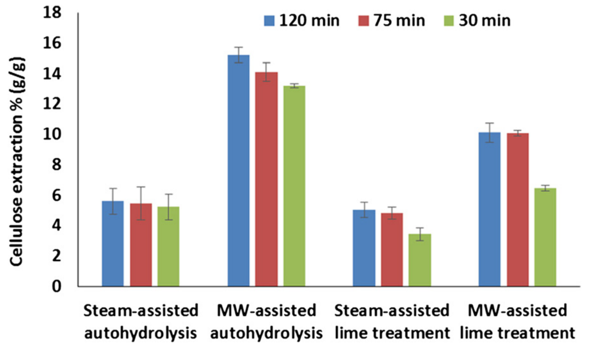

Fig. 6 Comparison of cellulose extraction during the steam-assisted and MW-assisted treatments of SSB (the *error bars* are showing the ±standard deviations)

The SALT resulted in less cellulose hydrolysed into the yellow liquor from SAHT-SSB than during the MALT from the MAHT-SSB. The quantities of cellulose hydrolysed during the SALT and the MALT were 3.90 (±1.45) % and 7.70 (±2.71) %, respectively. The difference in quantity of cellulose hydrolysed between the two treatments was found to be significant (p value <0.05). Under optimum conditions, the SALT of the autohydrolysed SSB resulted in 4.70 (±0.22) % cellulose hydrolysis, whereas under optimum conditions, the MALT of the MAHT-SSB resulted in 10.07 (±0.18) % cellulose hydrolysis. In comparison to the steam-assisted fractionation, the microwave-assisted fractionation process resulted in more cellulose hydrolysed from SSB. This can be attributed to the higher energy absorbed by the mixture during microwave treatment and the accompanied physicochemical changes to the cellulose component of SSB [36]. However, further studies are required to know the exact nature of the cellulose fractions dissolved during the MW-assisted treatments. The results have shown that the MW-treatments have specific effect on the cellulose component of SSB.

Comparison of steam-assisted versus microwave-assisted treatments on ash concentration of the sweet sorghum bagasse residues

SSB contained only 0.78 (±0.22) % ash in it. The hydrothermal treatments resulted in slight increase in the ash concentration because of the relatively high extraction of other biomass components from the substrate. The concentration of ash in the residue obtained from the SAHT and the MAHT were 1.01 (±0.02) % and 0.92 (±0.06) %, respectively. Under optimum conditions, the SAHT resulted in 1.01 (±0.01) % of ash concentration in the SAHT-SSB, whereas under optimum conditions for MAHT, the ash concentration in the SSB residue was found to be 0.88 (±0.01) %. The *t*-test results have proved that the steam-assisted and microwave-assisted hydrothermal treatments have no significantly different effects on the concentration of ash in the autohydrolysed SSB. It is also clear that the hydrothermal treatments do not significantly affect the ash content in the SSB [37].

The lime treatment resulted in increasing the ash content of the lime-treated SSB. The increase in ash concentration can be attributed to the binding of calcium ions to the carbohydrate and lignin components during the lime treatment [38]. The concentrations of ash in the residue of SALT and the residue of MALT were 2.62 (±1.38) % and 3.05 (±0.95) %, respectively. The difference in concentration of ash in the residues obtained after the two treatments was found to be not significant. Under optimum conditions, the SALT of the SAHT-SSB resulted in 4.25

(±0.39) % ash concentration in it, whereas under optimum conditions, the MALT of the MAHT-SSB resulted in 4.03 (±0.21) % ash concentration. The similar concentration of ash in the lime-treated residues has shown that, in comparison to the SALT the MALT, it does not affect the binding of calcium ions to the carbohydrates and lignin components in the SSB. However, the increased concentration of ash may have negative effects on further processing of the treated SSB.

Comparison of steam-assisted versus microwave-assisted treatments on isolation of lignin and lime from the yellow liquor

Maximum precipitation of lignin occurred at pH 4.5 (±0.25), and the CO_2 treatment resulted in 58.85 (±3.2) % recovery of lignin from the yellow liquor of the SALT. Similarly, the CO_2 treatment of the yellow liquor of MALT resulted in 60.26 (±2.11) % lignin precipitation at pH 4.5 (±0.28). There was no effect of the treatment applied to the SSB on the quantity of lignin recovered from the yellow liquor through the CO_2 addition because the difference in extent of the precipitation of calcium carbonate from the yellow liquors was found to be not significant. The quantity of precipitates obtained per litre of the yellow liquor of the SALT and the MALT was 3.02 (±1.34) g/L and 3.34 (±0.79) g/L, respectively. This was accounted for 26.57 (±8.68) % and 32.35 (±14.44) % removal of total dissolved solids (TDS) from the respective yellow liquors.

Maximum precipitation of $CaCO_3$ occurred at pH 9.5 (±0.06), and the CO_2 treatment resulted in 65.99 (±1.2) % recovery of lime from the yellow liquor of the SALT of the SAHT-SSB. Similarly, the CO_2 treatment of the yellow liquor of MALT resulted in 71.85 (±2.48) % precipitation of $CaCO_3$ at pH 9.5 (±0.10). There was no effect of the treatment applied to the SSB on the quantity of lime recovered from the yellow liquor through the CO_2 addition because the difference in extent of the precipitation of calcium carbonate from the yellow liquors was found to be not significant.

Comparison of steam-assisted versus microwave-assisted treatments on composition and properties of the products of sweet sorghum bagasse fractionation

The hydrothermal treatments of SSB resulted in a hydrolysate rich in reducing sugars and a solid residue rich in cellulose and lignin. During the hydrothermal treatment, hemicellulose was depolymerized into oligosaccharides, monosaccharides and sugar degradation products [3]. The reducing sugar concentration in the hydrolysate from the steam-assisted hydrothermal treatment was considerably less than the reducing sugar concentration in the hydrolysate from the MAHT (Fig. 7). The SAHT of SSB, at 10 % (g/g mixture) substrate concentration, resulted in 37.97 (±2.33) g/L reducing sugar concentration while the MAHT resulted in 56.85 (±3.84) g/L of reducing sugars in the hydrolysate. The difference in concentration of reducing sugars in the hydrolysates, over the range of treatment conditions studied, is found to be significant (*p* value <0.05), and the microwave treatment was found to be increasing the reducing sugar concentration in the hydrolysates from SSB. However, under the optimum conditions, the SAHT of SSB, at 13 % (g/g mixture) substrate concentration, resulted in 59.80 (±3.74) g/L reducing sugar concentration in the hydrolysate. Similarly, under the optimum conditions, the MAHT of SSB resulted in 60.82 (±3.69) g/L reducing sugars in the hydrolysate. In comparison to the SAHT, the increased reducing sugar concentration during the MAHT of SSB can be attributed to the increased hydrolysis of cellulose during the MAHT. The higher concentration of reducing sugar concentration will be useful for the economical production of value added products from the hydrolysate.

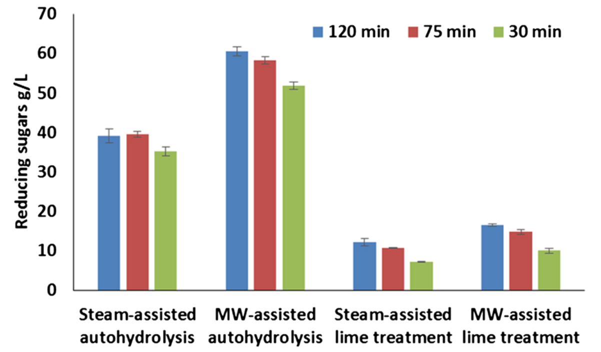

Fig. 7 Comparison of reducing sugar concentration in the liquid products of the steam-assisted and MW-assisted treatments of SSB (the *error bars* are showing the ±standard deviations).

In addition to the delignification, the lime treatment also caused the hydrolysis of a fraction of the hemicellulose and cellulose in the hydrothermally treated SSB. The reducing sugar concentration in the yellow liquor from the steam-assisted lime treatment was considerably less than the reducing sugar concentration in the yellow liquor from the MALT. The SALT of SAHT-SSB resulted in 8.59 (±3.49) g/L reducing sugar concentration while the MALT resulted in 13.50 (±3.75) g/L of reducing sugars in the yellow liquor. Under the optimum conditions, the SALT of the SAHT-SSB resulted in 12.12 (±0.24) g/L reducing sugar concentration in the yellow liquor, whereas under the optimum conditions, the MALT of MAHT-SSB resulted in 15.11 (±0.86) g/L reducing sugars in the yellow liquor. The difference in concentration of reducing sugars in the yellow liquor, over the range of treatment conditions studied, is found to be significant (*p* value <0.05), and the microwave treatment was found to be increasing the reducing sugar concentration in the yellow liquor from

MAHT-SSB. In comparison to the SALT, the increased reducing sugar concentration during the MALT of MAHT-SSB can be attributed to the increased hydrolysis of hemicellulose and cellulose during the MALT.

The concentrations of total carbohydrates, pentose sugars, furfurals and 5-hydroxy methyl furfurals in the hydrolysates and yellow liquors produced by the steam-assisted and MW-assisted treatments were significantly different (*p* value <0.05), and the pattern in their concentration was like that of reducing sugars.

The HT-SSB contained higher concentration of cellulose and lignin than the untreated SSB. The chemical composition of the SAHT-SSB, produced under the optimum treatment conditions, was 56.36 (±0.06) % cellulose, 11.11 (±0.01) % hemicellulose, 31.42 (±0.01) % lignin and 0.99 (±0.01) % ash, whereas the MAHT resulted in a residue with 53.38 (±0.14) % cellulose, 12.72 (±0.18) % hemicellulose, 30.19 (±0.47) % lignin and 0.88 (±0.01) % ash in it.

The MALT-SSB contained higher concentration of cellulose than the SALT-SSB samples. The chemical composition of the SALT-SSB, produced under the optimum treatment conditions, contained 68.29 (±0.31) % cellulose, 10.77 (±0.15) % hemicellulose, 13.26 (±0.36) % lignin and 4.25 (±0.39) % ash, whereas the MALT resulted in a residue with 69.41 (±1.83) % cellulose, 12.14 (±0.71) % hemicellulose, 12.59 (±0.31) % lignin and 4.03 (±0.21) % ash in it. The difference in composition of the SSB samples treated using the steam-assisted and MW-assisted processes was found to be not significant. Both the steam-assisted treatment and the MW-assisted treatments have similar effects on the fractionation of SSB and the composition of the residual SSB.

Comparison of the FTIR spectra of the SSB samples and lignins produced through the steam-assisted and microwave-assisted treatments

The FTIR spectra of the SSB samples which have undergone steam-assisted and MW-assisted treatments have shown (Fig. 8) characteristic absorption at wavenumbers specific for cellulose (1428 cm^{-1}), hemicellulose (1371 cm^{-1}) and lignin (1510 cm^{-1}). The increase and decrease in absorption intensities at these wavenumbers were corresponding to the treatments received by the SSB. Hydrothermal treatments decreased the concentration of hemicellulose in the biomass, and it resulted in decrease in absorption intensities at 1371 cm^{-1}.

Likewise, hydrothermal treatments lead to increase in concentration of lignin and cellulose, and the intensity of absorption at 900 and 1428 cm^{-1} by cellulose and 1510 cm^{-1} by lignin was increased in comparison to the untreated SSB. Intensity of absorption at 1428 cm^{-1} increased further with decrease in the intensity of absorption at 1510 cm^{-1} in the two-step treated SSB confirming the increase in the concentration of cellulose and decrease in the concentration of lignin.

The spectra of hydrothermally treated SSB samples produced through the steam-assisted and MW-assisted treatments have shown variations in the intensities of absorption at 900 and 1428 cm^{-1} confirming the variation in the concentration and nature of cellulose [39]. Intensity of absorption at 900 cm^{-1} by cellulose in SAHT-SSB was higher than by the cellulose in MAHT-SSB showing the presence of more amorphous cellulose in the former. The higher intensity of absorption at 1428 cm^{-1} by the cellulose in MAHT-SSB than by the cellulose in SAHT-SSB shows the presence of more crystalline cellulose in the former. The total crystallinity index (TCI) was 0.46 for the SAHT-SSB and was 1.22 for the MAHT-SSB. Thus it is clear that the MAHT produced more crystalline cellulose than the SAHT in the SSB [39].

The concentration of hemicellulose in the MAHT-SSB was slightly higher than in the SAHT-SSB, and it was confirmed by the relatively high intensity in absorption at 1371 cm^{-1} by the hemicellulose in the former. Similarly, the intensity in the absorption at 1510 cm^{-1} by the lignin component of the SAHT-SSB was higher than by the lignin in the MAHT-SSB confirming the high concentration of lignin in the former [39].

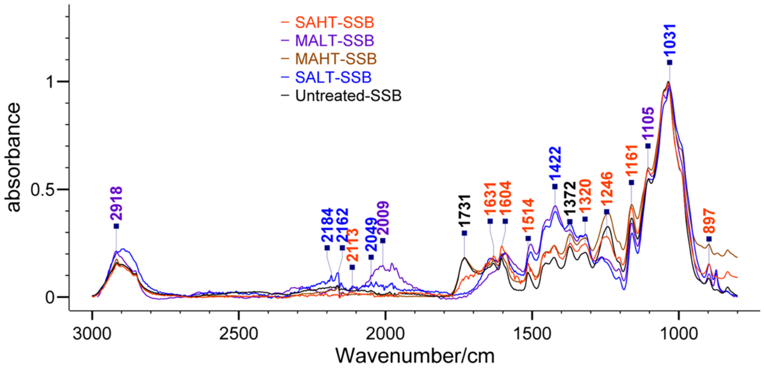

Fig. 8 Comparison of FTIR spectra of SSB samples produced through the steam-assisted and MW-assisted treatments

The cellulose concentration in the SSB samples produced by the MALT was slightly higher than in the SSB samples produced by the SALT. The intensity of absorption at 1428 cm^{-1} by the cellulose in SSB sample produced by the MALT was higher than by the cellulose in the SSB sample produced by the SALT. The two-step treatment increases the TCI values of SSB samples produced by the steam-assisted and MW-assisted treatments, and the latter process produced a sample with TCI value of 2.83. Similarly, the TCI value of the SSB samples produced through the SALT of the SAHT-SSB was 1.70. Thus, it is clear that the MW-assisted treatments can be used for the production of crystalline cellulose from lignocellulosic materials. Crystalline cellulose can be used to produce high-value products such as bio-nano-composites having many industrial applications in agriculture, environment, packaging, textiles, etc. [40, 41].

The intensity of absorption at 1500–1600 cm^{-1} was reduced in the lime-treated SSB samples from that of untreated and HT-SSB samples, confirming the extraction of lignin component from them through the lime treatment. The intensity of absorption was higher in the SSB samples produced through the MALT than by the SALT. It confirms the relatively higher concentration of lignin in the SSB samples produced through the SALT than by the MALT [42].

The FTIR spectra of the samples of precipitated lignin from the yellow liquors of the SALT and MALT have shown slight variation in the intensities of absorption at their characteristic wavenumbers (Fig. 9). The intensity of absorption at 1510 cm^{-1}, characteristic of the carbonyl group was higher for the lignin precipitated from the yellow liquor of the MALT. The higher absorption at 1200–1220, 1265 and 1510 cm^{-1} by the lignin of MW-assisted process than by the lignin of the steam-assisted process was from the relatively higher concentration of guaiacyl lignin in the precipitate. The absorption peak at 1315–1321 cm^{-1} which is characteristic of the syringyl lignin group was absent in both the precipitates [39, 43]. The presence of higher concentration of guaiacyl groups in the precipitates can be attributed to the extraction of both syringyl and guaiacyl lignin groups by the lime treatments and precipitation of mostly the guaiacyl groups in the acidic conditions [44].

Comparison of the DSC thermograms of the SSB samples and lignins produced through the steam-assisted and microwave-assisted treatments

The DSC thermograms of the SSB samples treated with the two methods have shown differences in the specific temperatures for thermal reactions and the accompanied degradation (Fig. 10). The samples were not completely decomposed at 500 °C, and higher temperature was required for the complete decomposition. All samples have shown endothermic and exothermic changes while heating from 25 to 500 °C. The temperature at which the endothermic and exothermic changes exhibited was varying from sample to sample. The first endothermic change around 90–120 °C can be attributed to the loss of moisture, and depending on the composition of the material, the evaporation occurs over a wide range of temperature [45]. The SAHT-SSB and the MAHT-SSB exhibited high exothermic reactions at around 420 and 400 °C, respectively, which can be attributed to the decomposition of cellulose. Similarly, the exothermic peak at around 320 °C by the SAHT-SSB and the MAHT-SSB can be attributed to the decomposition of hemicellulose remaining in them. The MAHT-SSB exhibited relatively less exothermic heat flow than the SAHT-SSB, and it can be attributed to the relatively less concentration of lignin in the former as described by Tsujiyama and Miyamori [46].

The two-step treated SSB samples from both the methods exhibited difference in thermal decomposition, and the variation can be because of the nature of cellulose component in them. The SSB sample produced through the MALT has more crystalline cellulose when compared to the SSB sample produced through the SALT. The former sample exhibited higher exothermic reactions within the temperature studied [47].

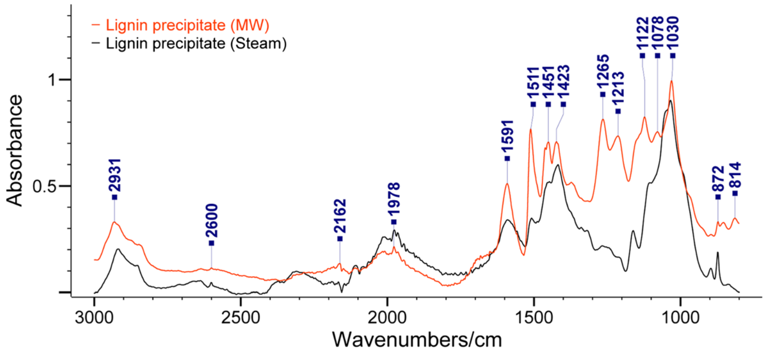

Fig. 9 Comparison of FTIR spectra of lignin samples produced through the steam-assisted and MW-assisted treatments

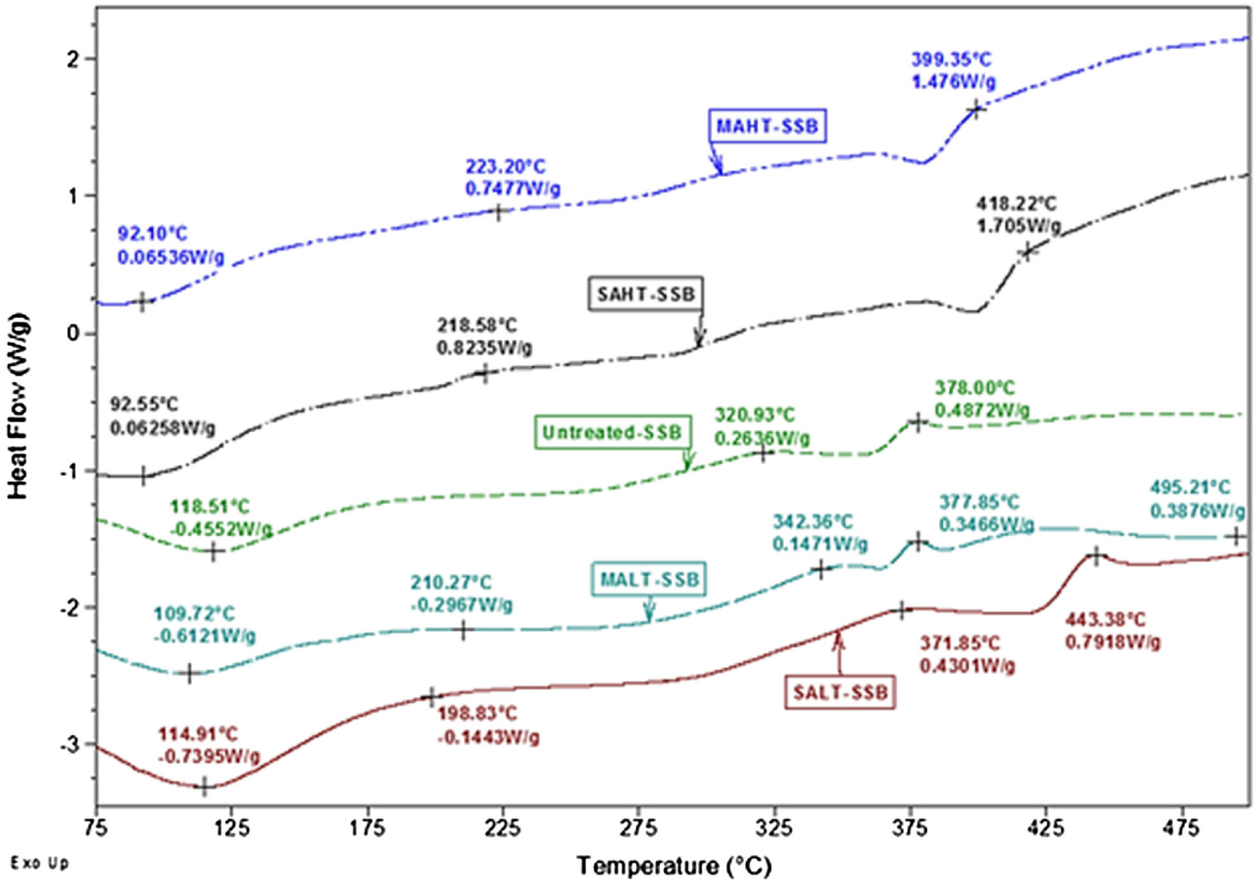

Fig. 10 Comparison of the DSC thermograms of the SSB samples produced through the steam-assisted and MW-assisted treatments

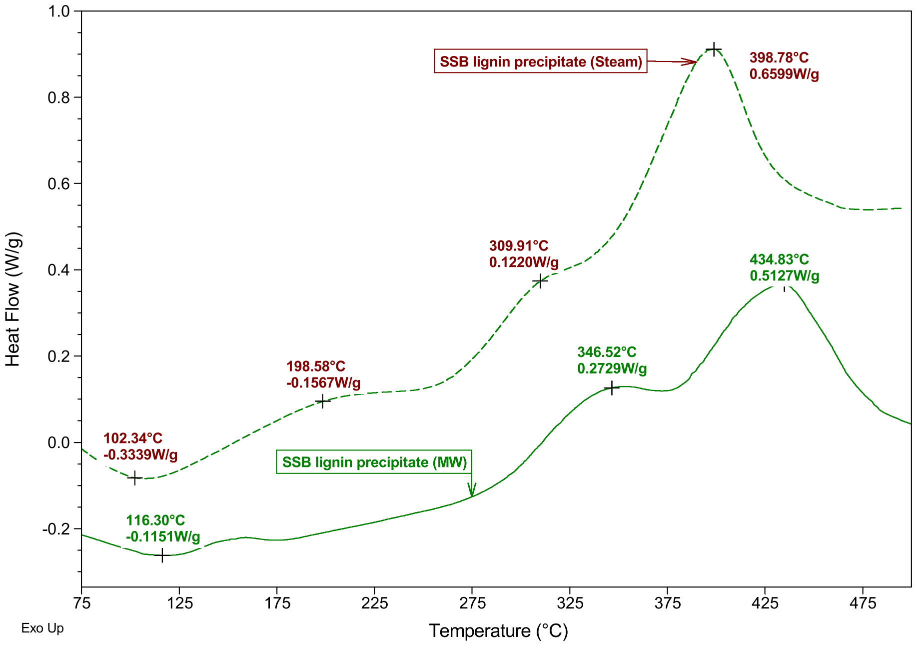

Fig. 11 Comparison of the DSC thermograms of the lignin samples produced through the steam-assisted and MW-assisted treatments

The thermograms of the precipitated lignin samples have shown differences in their thermal decomposition (Fig. 11). The precipitated lignin was thermally less stable than the lignin in the SSB. It can be attributed to the presence of impurities such as carbohydrates and minerals in the precipitated lignin [46]. Both the lignin precipitates were dehydrated in the range of 100–120 °C. The lignin precipitated from the yellow liquor of the MW-assisted process was more thermally stable and decomposed in the range of 425–445 °C while the lignin from the steam-

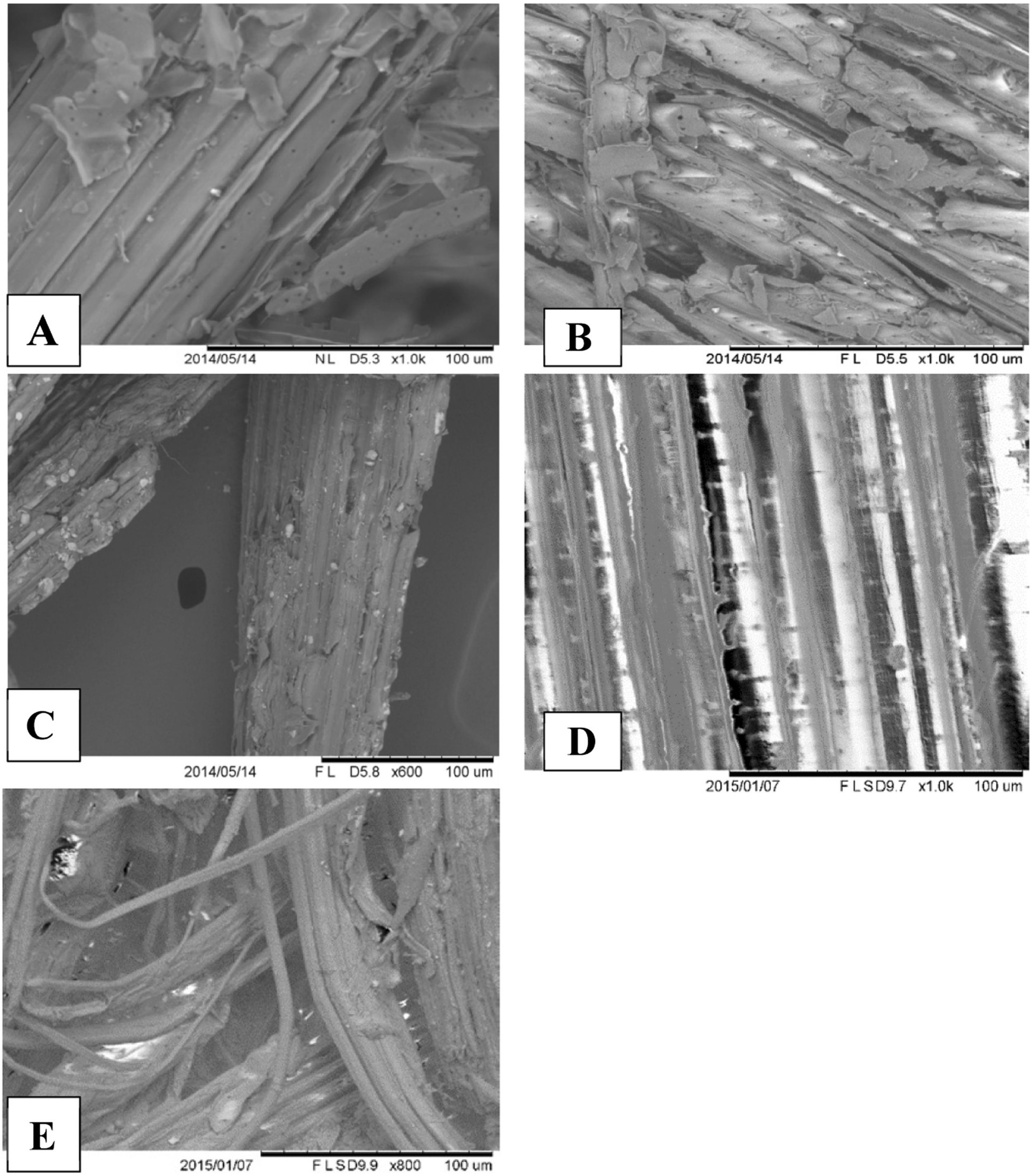

Fig. 12 SEM images of the SSB samples produced though the steam-assisted and MW-assisted treatments (**a** untreated-SSB, **b** SAHT-SSB, **c** SALT-SSB, **d** MAHT-SSB and **e** MALT-SSB)

assisted process decomposed in the range of 380–400 °C. The exothermic shoulder in the range of 310–350 °C in the lignin samples can be attributed to the presence of carbohydrate compounds such as cellulose [45].

Comparison of the SEM images of the SSB samples produced through the steam-assisted and microwave-assisted treatments

The scanning electron microscope (SEM) images of the treated samples were compared with each other to observe the morphological changes on them (Fig. 12). The surface of the untreated SSB has shown the breakage from the mechanical size reduction. In comparison to the untreated SSB, the hydrothermally treated and lime-treated SSB samples have more pores and crevices on their surface. The HT-SSB samples have a shiny surface from the condensation of lignin. The surface of the MAHT-SSB was shinier than the surface of the SAHT-SSB. The fibers were seen as adhered on the surface which can be attributed to the condensation of furfural with the lignin [48]. The surface of the lime-treated samples appeared as coarser than the hydrothermally treated and untreated SSB samples. It can be attributed to the removal of the lignin binding the fibers in the substrates. The MALT has separated the fibers on the MAHT-SSB, and the separation was less observed on the surface of SALT-SSB. The increased separation can be because of the increased extraction of lignin by the MALT. Calcium carbonate crystals are visible on the surface of both the SSB samples produced through the two methods compared. The increased surface area of the MALT-SSB can enhance the accessibility for the enzymes during the hydrolysis stages [49].

Comparison of steam-assisted versus microwave-assisted treatments for the scope of on-farm fractionation of SSB

Steam-assisted processing has the potential to be applied for the on-farm processing of biomass to produce intermediary products for biorefineries and other industries. Researchers have also considered the use of microwave reactors for the on-farm processing of biomass to reduce the transportation cost [50].

The steam-assisted and MW-assisted fractionation processes evaluated in this study have produced a hydrolysate containing pentose sugars, a solid residue rich in cellulose, and solid lignin as products from the SSB. The bulk density of the solid residue obtained after the SALT was about 3.7 times more than that of the raw bagasse. Similarly, the bulk density of the solid residue obtained after the MALT was about 3.6 times more than that of the untreated SSB. The increased bulk density would make the transport of the two-step treated SSB to the distant biorefineries more economical [51].

An overall comparison of the important outcomes of the fractionation of SSB using the steam-assisted and MW-assisted processes is shown in Table 3. The steam-assisted process produces a solid residue with slightly higher bulk density and lignin content than the MW-assisted process. The MW-assisted process produces a hydrolysate containing slightly higher reducing sugars and a solid residue containing slightly higher cellulose content than the steam-assisted process. The MW-assisted process takes 27 % less time than the steam-assisted process to achieve the similar fractionation results for the SSB. The selection of the process for the on-farm processing can only be made after considering the technical, economic and environmental aspects of the two processes especially the capital investment required for each of the processes [52].

Conclusions

The steam-assisted and MW-assisted processes fractionated the SSB into hemicellulosic sugars, cellulose-rich and high-density residue and solid lignin. The processes also recovered a part of the lime used for the fractionation of SSB. The two methods have not exhibited significant differences in overall recovery of the solids, hemicellulose extraction, delignification, residual concentration of cellulose and ash, or in the recovery of lignin and lime. The difference was significant only in the concentration of total reducing sugars in the hydrolysate and the yellow liquor. The MW-assisted process increased the crystallinity of the cellulose in the treated SSB and also increased the concentration of guaiacyl lignin content in the recovered lignin which was thermally more stable than the lignin produced in the steam-assisted process. The steam-assisted process produced denser SSB which was thermally more stable than the SSB produced through the MW-assisted process. The MW-assisted process resulted in increased separation of the fibers in the SSB than by the steam-assisted process. The techno-economic and ecological analyses of the two

Table 3 An overall comparison of the process results of steam-assisted and MW-assisted fractionation of SSB

Process result	Steam-assisted	MW-assisted
Solids recovery (%)	57.84 (±0.09)	52.04 (±0.76)
Reducing sugars in the hydrolysate (%)	22.62 (±1.10)	27.10 (±0.11)
Cellulose concentration in the residue (%)	68.29 (±0.31)	69.41 (±1.83)
Bulk density of the residue (kg/cm^3)	314.5 (±11.37)	304.33 (±15.50)
Lignin recovery (%)	41.14 (±1.04)	40.05 (±0.65)
Lime recovery (%)	65.99 (±1.2)	71.85 (±2.48)
Isothermal treatment residence time (min)	196	143

processes need to be conducted to assess their potential for the on-farm applications.

Competing interests

The authors declare that they have no competing interests.

Authors' contributions

JKK conducted the experiments, analysed the results and prepared the manuscript. YG has provided the technical support for the experiments. VO facilitated the FTIR studies and also provided technical suggestions on the study. GSVR supervised and guided the research work. All authors have read and approved the manuscript.

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