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Pretreatment of Crop Residues by Application of Microwave Heating and Alkaline Solution for Biofuel Processing: A Review

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Abstract

The effect of microwave-assisted alkaline pretreatments and enzymatic saccharification of lignocellulosic agricultural crop residues are reviewed. Pretreatment is a major step for the efficient and effective biochemical conversion of lignocellulosic biomass to biofuel. Microwave-assisted alkali pretreatment is one of the promising techniques used in the bioconversion of biomass into useful energy product. The advantages of microwave heating coupled with alkaline pretreatment include reduction of the process energy requirement, rapid and super heating, and low toxic compound formation. This chapter reviews recent microwave-assisted alkali pretreatment and enzymatic saccharification techniques on different agricultural residues highlighting lignocellulosic biomass treatments and reducing sugar yields, and recovery. In addition, compiled up-to-date research studies, development efforts and research findings related to microwave-assisted alkali, and enzymatic hydrolysis are provided.

Keywords: microwave, Lignocellulosic biomass, agricultural residues, enzymatic saccharification, glucose yield

1. Introduction

Lignocellulosic biomass feedstock is one potential source of renewable energy and considered as a non-food material (second-generation feedstock) [1]. Agricultural and forest residues as well as industrial and municipal solid wastes are made up of lignocellulosic components [2]. They are environmentally friendly with a carbon-neutral footprint when converted to renewable

energy, compared to fossil energy sources such as crude oil, coal, and natural gas [3]. Lignocellulose biomass consists of cellulose, hemicellulose, and lignin. Both cellulose and hemicellulose are polysaccharides with cellulose being the main molecule utilized for ethanol production. Unlike cellulose, which comprises long unbranched fibrils entirely made up of glucose, hemicellulose is a branched polymer, and its polymer chains are shorter than those of cellulose which are described as water soluble because some sugar units are linked to the acetyl groups [4–6]. Lignin acts as a glue between hemicellulose and cellulose and still has some energy value, which can be converted to a variety of value-added products [7, 8].

The production of bioethanol from lignocellulosic biomass (crop residues and waste crops) has been estimated to be 422–491 billion liters per year, which is 16 times higher than global bioethanol production [9]. Bioethanol blend with gasoline (E5, E10, and E85) indicates greenhouse gas (GHG) emission advantage since bioethanol is less carbon-rich than gasoline [10, 11]. The lignocellulosic bioethanol process can be categorized into four steps: pretreatment, saccharification, fermentation, and product (ethanol) recovery [12, 13]. Pretreatment facilitates the breakdown of cell walls and internal tissues of the lignocellulosic biomass through physical, chemical, and biochemical conversion processes. This process involves the disruption and disintegration of recalcitrant structures to open channels for enzymatic reactions in the substrate [14–16]. According to the U.S. Department of Energy [17], the biomass process dramatically reduces dependence on crude oil, supports the use of diverse, domestic, and sustainable energy resources, provides a basis for bioindustry development in accelerating economic growth, and represents an effective strategy for reducing carbon emissions from energy production and consumption.

Recently, many research works have described pretreatment as the most expensive stage in bioethanol production considering challenges faced during the conversion process [18]. Pretreatment accelerates lignocellulosic solubilization, thereby improving enzymatic reactions in the material [12, 15, 16]. **Figure 1** shows a schematic of the effect of pretreatment on lignocellulosic biomass. An effective pretreatment technique is needed to liberate the cellulose from lignin, reduce cellulose crystallinity, and increase cellulose porosity [11]. Various pretreatment methods have been developed according to different research studies [3], but the choice of pretreatment technique for a raw material/feedstock is influenced by many factors. These include sugar recovery yield, low moisture content effectiveness, lignin recovery, required particle size, and low energy demand [20].

Pretreatment methods include microwave (MW)-assisted, dilute acid, alkali, steam explosion, ammonia fiber explosion (AFEX), lime, organic solvent, ionic liquids, and biological. A combination of these methods has also been studied, and some studies are still ongoing [3, 20]. Kumar et al. [21] and Merino-Perez et al. [22] presented advantages of pretreatment on lignocellulosic biomass such as (1) improved substrates sugar formation, (2) avoid degradation of carbohydrate, (3) avoid the generation of toxic compounds that can inhibit hydrolysis and fermentation processes, (4) avoid the decomposition of cellulose and hemicellulose, (5) reduction in the number and quantity of chemical reagents used, and (6) cost-effectiveness. Many research reports have compared various pretreatment methods of lignocellulosic biomass, indicating advantages and disadvantages of each pretreatment method. However, the choice

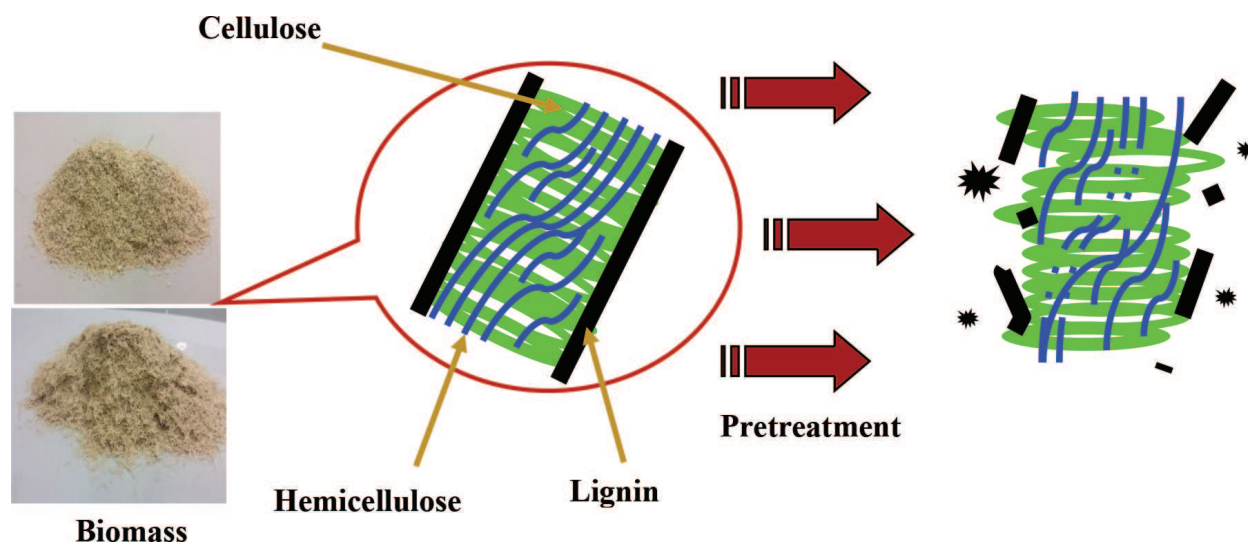


Figure 1. Effect of pretreatment on biomass [19].

of each pretreatment technique depends on the type of biomass, composition, and resulting byproducts [23, 24]. In addition, most of the pretreatment techniques suffer relatively low sugar yields, severe reaction conditions, high processing costs and capital investment, and investment risk [25]. Research efforts are continuing to address these challenges. For instance, there is growing interest in microwave heating as a pretreatment alternative to support second-generation lignocellulosic biorefineries. According to Aguilar-Reynosa et al. [18], microwave heating process has attracted a series of experimental techniques because it satisfies green chemistry, reduction of time of processing by 10 times compared to other heating techniques, fast heat transfer, and essentially an alternative method to conventional heating [26–28].

This review is intended to identify the various microwave-assisted alkali pretreatment effects on the different lignocellulosic agricultural residues. Emphasis is also placed on the pretreatment process effects of the lignocellulosic biomass and its sugar yield/recovery from enzymatic saccharification.

2. Microwave heating effects

Microwave (MW) irradiation refers to electromagnetic waves that consist of electric and magnetic fields. The waves are formed within a frequency band of 300 MHz and 300 GHz [29, 30]. The operational frequency of a domestic microwave oven is 2450 MHz and its heating mechanism with a material depends on shape, size, dielectric constant, and the nature of the microwave equipment. The heating mechanism in MW is aligned with dipolar polarization, conduction, and interfacial polarization. The alignment of polar molecules in an electromagnetic wave with rapid oscillation caused by microwave irradiation forces the polar molecules to align in the radiation field. Dipolar polarization is responsible for the continuous alignment of the polar molecules inside the material which generates the heat [30, 31]. Motasemi and

Afzal [32] and Xu [31] described three ways in which MW irradiation and materials can interact, namely (1) MW-transparent material (insulator) where microwaves pass through without losses like Teflon™ or quartz, (2) conducting material which cannot allow microwave penetration but reflected like metals, and (3) absorbing materials such as oil and water. The electromagnetic radiation in MW heating is shaped like energy propagating in a vacuum without any material in motion, can be observed as light, and used as waves and non-ionizing waves in mobile cell phones and infrared [33].

In 1949, Spencer Percy discovered that electromagnetic frequency radiation could be used in dielectric heating via microwave for heating application in food and other process requiring the use of heat. Von Hippel in 1954 provided further elaboration based on understanding of theories on macroscopic interactions of microwave and matters explaining his theory with dissipated power, electric field intensity, and propagation constant [18]. MW heating is directly from inside the material – wave interactions, leading to heat transfer and basically has a higher energy yield in comparison with conventional oven techniques which transmit heat by conduction-convection mechanism [34]. Xu [31] stated that conventional heating is transferred from the surface toward the center of the material by conduction, convection, and radiation; however, MW heating converts electromagnetic energy into thermal energy.

Numerous research studies have reported advantages and disadvantages of MW relative to conventional heating [18]. Advantages of MW include shorter residence time, faster heat transfer, selective, instantaneous on and off operation, precise and controlled heating, rapid and efficient, and environmental friendly process [4]. Due to its efficient process, MW heating has limited disadvantages such as (1) poor distribution of MW power within the material because of non-homogeneous material, (2) non-uniform heating, and (3) low penetration of radiation in bulk materials [35–37].

2.1. Alkali pretreatment of lignocellulosic biomass

Alkaline pretreatment improves cellulose digestibility, the ability to saponify intermolecular ester bonds, cross-linking xylan hemicelluloses, and other components. The effect of alkali pretreatment on lignocellulosic biomass is dependent on lignin content [20]. Alkaline reagents suitable for alkali pretreatment are NaOH, KOH, $\text{Ca}(\text{OH})_2$, and NH_4OH . The sugar yield of alkali pretreatment is dependent on the feedstock used [3]. Some of the alkalis cause swelling, an increase in cellulose internal surface area, decreasing the degree of polymerization and crystallinity [38], while some disrupt the lignin structure of the material and remove acetyl groups from hemicellulose, thereby enhancing cellulose digestibility and increasing the reactivity of the remaining polysaccharides during delignification [12, 15, 22]. The advantages of alkali pretreatment are no washing of samples after pretreatment, no corrosion problem in the equipment used for the treatment as compared to acid, and the use of lower temperatures and pressures compared with other pretreatment techniques [39, 40].

2.2. Microwave-assisted alkali pretreatment technology and enzymatic saccharification

Ethanol from cellulose-based biomass is one of the most attractive alternatives to replace fossil fuels because using non-edible material as feedstock to produce ethanol and corresponding

bioproducts minimizes environmental challenges [7, 22]. Microwave pretreatment method is a physico-chemical process involving thermal and non-thermal effects. The early discoveries of microwave pretreatment on lignocellulosic biomass were reported by Ooshima et al. [41] and Azuma et al. [42]. Since then, the technology has shown efficient applications in various ways [43–45]. Recently, many research studies have used MW heating as a pretreatment technique to assist in converting lignocellulosic biomass into useful bioproducts [46]. MW pretreatment combines both thermal and non-thermal effects within the aqueous environment of physical, chemical, or biological reactions [47], and its thermal heating may considerably decrease the time and efficiency of the pretreatment [22]. The pretreatment of lignocellulosic biomass using MW heating is done selectively especially at the polar parts, resulting in an increase in the disruption of the recalcitrant structures of the biomass [48]. To date, different pretreatment techniques to make lignocellulose accessible to enzymes for enhancing bioethanol conversion have been widely studied [20]. On the other hand, energy utilization in the pretreatment process raises the overall cost of producing bioethanol considerably, and this is a critical factor to consider before investing in biorefinery processes [20]. Darji et al. [49] and Aguilar-Reynosa et al. [18] reported the different studies on MW heating process describing MW heating as a better technology with energy efficiency to reduce energy consumption during pretreatment.

Enzymatic saccharification is a biochemical conversion preceded by pretreatment and followed by microbial catalyst conversion [50]. This is a microbial degradation process, accomplished by using enzymes and the result is usually a decrease in sugar [12]. Converting lignocellulosic biomass to ethanol involves disintegrating the biomass cell wall structure, thereby releasing the simple sugars which are fermented by yeast to produce ethanol [51]. Maitan-Alfenas et al. [52] reported that microorganisms are essential in enzyme production for lignocellulosic biomass saccharification. The saccharification process in the ethanol conversion requires less energy and is done in mild conditions at pH of 5.2–6.2 and a temperature range of 45–50°C [53, 54]. There are three distinct major types of cellulase enzymes used in the process: (1) endoglucanases (E C 3.2.1.4) hydrolyze at random internal β -1, 4-glucosidic linkages in the cellulose chain producing oligosaccharides of different lengths and with a shorter chain appearance; (2) exoglucanases of cellobiohydrolases (E C 3.2.1.91) progress along cellulose chain ends and release major products as cellulose or glucose; and (3) β -glucosidases known as β -glucoside glucohydrolases (E C 3.2.1.21) hydrolyze cellulose to glucose, liberate cellobiose, soluble cellodextrins to glucose [12, 55]. For hemicellulases, hydrolysis of the hemicellulose fraction requires more complex group of enzymes, and endo- β -1, 4-xylanase enzyme is needed for the hydrolysis of xylana, the major polymer component in hemicelluloses [56, 57]. Cellulases and hemicellulases production involve many microorganisms such as filamentous fungi (*Trichoderma* spp. and *Aspergillus* spp. native or genetically modified). During saccharification process, one of the fungi lacks β -glucosidase activity (*Trichoderma*), and it is supplemented with *Aspergillus* spp. in enzymes blending to improve the conversion of lignocellulose to simple sugars [9, 52, 58, 59].

Related research investigations have reported different activities of enzymatic saccharification process with limiting factors on the lignocellulosic biomass such as moisture, available surface area, crystallinity of cellulose, degree of polymerization, and lignin content [8, 20, 60, 61]. Biomass formed in a complex network of lignocellulose contents has indicated that most enzymes used in process can be absorbed by resultant condensed lignin to reduced yield by

nonspecific linkages of the enzymes [52]. In addition, Palonen et al. [62] reported that the hemicellulose removal increases the mean pore size of the biomass, thereby increasing the chances of cellulose to get hydrolyzed. Consequently, lignin content reduces enzymatic saccharification by forming a shield and blocking substrate digestible parts from hydrolyzing [60]. Janker-Obermeier et al. [63] studied solubilization of hemicellulose and lignin from wheat straw through MW-assisted alkali treatment. The result suggested that more than 80% hemicellulose and 90% lignin could be removed from the solid wheat straw substrate without excessive saccharide solubilizing high amount of cellulose.

The combination of MW-assisted pretreatment and chemical pretreatment on different biomass as reported by several research studies indicated a higher sugar recovery, and various chemicals used in this process are dilute ammonia, iron-chloride and the common ones, alkaline and acid. All these chemicals assist MW pretreatment technology in removing lignin (alkali solution) and hemicellulose (acid solution) for cellulose accessibility [47]. The combined process separates lignocellulosic biomass components by disrupting the biomass structure, reducing the crystallinity of cellulose, improving the formation of fermentable sugars, and reducing the degradation of carbohydrates [64]. At lower temperatures, the combined pretreatment of lignocellulosic biomass improves enzymatic saccharification by accelerating the pretreatment reaction [65–67]. A combination of acid (H_2SO_4 , 2% w/v) and steam (140°C , 30 min) is reported to have efficiently solubilized the hemicellulose, resulting in 96% yield of pentose in pretreatment and enzymatic hydrolysis of soybean hull [68]. Consequently, more research studies on MW pretreatment technique are still ongoing using different feedstocks and chemical combinations.

2.3. Effect of microwave power, alkali concentration, and residence time on enzyme digestibility

MW-assisted alkali pretreatment can penetrate the biomass and vibrate the molecules. The rapid oscillation of the molecules causes continuous heat generation and disruption of lignocellulosic structure, and similar results were reported by Hamzah et al. [69] and Wang et al. [70]. **Table 1** describes the effect of microwave-assisted alkali technology in enhancing enzymatic saccharification. Lignocellulosic biomass conversion to bioethanol is very challenging considering the heterogeneous nature of the feedstock used in the process [22]. MW pretreatment process leads to a high lignin removal and improvement in the biomass morphology to facilitate the reactivity of the enzyme, thereby increasing sugar yields [22, 79]. Increasing the alkali concentration during pretreatment of lignocellulosic biomass helps to increase cellulose digestibility and is more effective for lignin solubilization [3, 48]; a relatively long residence time is needed to produce high sugar yields at a lower temperature for alkali pretreatment technique [3]. Increasing NaOH solutions (2–5 wt%) with high temperature (60 – 140°C) and residence time (10–60 min) at a fixed MW power level of 500 W dissolves a high amount of hemicellulose in the supernatant. Xylan recovery was 73%, and solubilization of lignin was highly dependent on the MW energy input [63]. Xu [31] reported that MW irradiation is an effective heat source employed in alkali pretreatment to produce temperature needed in the delignification of biomass for enzyme reactivity. However, MW-assisted alkali

Biomass	MW power (W)	MW time (min)	Alkali solution (%w/v)	Enzymes	Sugar analysis method	Sugar yield (dry biomass)	Reference
Oil palm empty fruit bunch (EFB)	180	3–21	NaOH	<i>Trichoderma reesei</i> and β -glucosidase	DNS	178 mg	[11]
Switchgrass and Coastal Bermudagrass	250	5–20	NaOH, Na ₂ CO ₃ and Ca(OH) ₂	<i>Trichoderma reesei</i> and β -glucosidase	DNS	2% NaOH/10 min: 82 and 63% glucose and xylose 1% NaOH/10 min: 87 and 59% glucose and xylose	[44]
Corn straw and rice husk	1300	2	Glycerol-water and glycerol-NaOH	<i>M. heterothallica</i> and cellulase Celluclast	DNS	Glycerol-NaOH: highest sugar yield for both samples	[71]
Switchgrass	1000 (Setting #1 to #4)	30–120 s	NaOH and water	Celluclast 1.5-L and Novozyme 188	NREL	0.1 g/g NaOH/30 min: 58.7 g/100 g Water/MW: 34.5 g/100 g	[66]
Sugarcane bagasse	100, 180, 300, 450, 600, and 850	1–30	NaOH and H ₂ SO ₄	Commercial cellulase	DNS	MW-alkali/600 W/4 min: 0.665 g/g MW-acid/100 W/30 min: 0.249 g/g MW-alkali (1%)-acid (1%): 0.83 g/g	[72]
Sweet sorghum bagasse (SSB)	1000	2, 4, 6	Lime	Cellulase (ACCELLERASE 1500)	DNS	SSB/MW/no-lime: 65.1% SSB/MW/lime: 52.6%	[73]
Wheat straw ear	400, 700, 1000	5–15 (5 min interval)	NaOH	Cellubrix L	NREL	1000 W/15 min: 148.93 g/kg Untreated: 26.78 g/kg	[74]
Switchgrass	250	5–20 (5 min interval)	NaOH and H ₂ SO ₄	<i>Trichoderma reesei</i> and β -glucosidase	DNS	NaOH/250 W/10 min: highest yield	[67]
Sweet sorghum juice	100–160 °C (15 °C interval)	60	Dilute ammonia	Spezyme CP and <i>Saccharomyces cerevisiae</i> (D ₅ A).	NREL	4.2 g glucose/10 g at low temperature and short MW time	[25]
Rice straw	70–700	1–5	NaOH	E-CLEAN, endo-1, 4- β -glucanase and EBLUC and β -glucosidase	DNS	MW-assisted alkali: 1334.79 μ g/ml	[75]
Pineapple	170–510 (170 W interval)	5, 10, 20, 40, 60, 120, 180 s	NaOH	Cellulase	HPAEC-PAD	33.5% total sugar yield at 6.375 W/g for 5 s	[76]
Cashew apple bagasse	600 or 900	15 or 30	NaOH	Commercial celluclast and β -glucosidase	NREL	0.2 and 1.0 mol/L NaOH: 372 and 355 mg/g	[77]

Biomass	MW power (W)	MW time (min)	Alkali solution (%w/v)	Enzymes	Sugar analysis method	Sugar yield (dry biomass)	Reference
Canola straw and Oat hull	713	6–18 (3 min interval)	NaOH and KOH	<i>Trichoderma reesei</i> and β -glucosidase	DNS	Canola straw: 110.0 mg/g Oat hull: 99.10 mg/g	[3]
Catalpa sawdust	200, 400, 600	3, 6, 9	MW-water, NaOH and Ca (OH) ₂	Commercial cellulase	DNS	MW/Ca(OH) ₂ /400 W/ 6 min: 402.73 mg/g	[78]

HPAEC-PAD: high-performance anion exchange chromatography-pulsed amperometric detector; DNS: dinitrosalicylic acid; NREL: National Renewable Energy Laboratory

Table 1. Summary effect of microwave power, residence time, and alkali concentration in improving enzymatic digestibility in selected agricultural crop residues.

pretreatment technology is effective depending on the lignin content of the feedstock and can lead to a high lignin solubilization and increased sugar yields [52]. According to Chaturvedi and Verma [79], results from reducing sugar yields ranging from 40 to 60% are mostly reported from MW-assisted pretreatments. The review pointed out that no pretreatment technology offers 100% conversion of biomass into fermentable sugars. To obtain the optimal MW-assisted alkali pretreatment condition that can improve enzymatic digestibility using different biomass, various microwave power levels, residence times, and alkali solutions of various concentrations were considered. Also, feedstock properties and reaction conditions are contributing factors influencing microwave pretreatment characterization and yield of the final product.

The results from **Table 1** indicate that MW-assisted alkali pretreatment can enhance the acceleration of enzymatic hydrolysis process compared to the conventional method as reported by many research studies. Sodium hydroxide (NaOH) solution identified as the most widely applied in MW pretreatment process and effective alkali compared to other alkalis. It was observed that NaOH, residence time, and substrate concentration were the main factors affecting the enzymatic saccharification efficiency. From the different MW-assisted alkali pretreatment processes, a low MW power (200–400 W) and a short exposure time (1–25 min) of feedstock reactor improved enzymatic saccharification sugar yields. However, lime was not a good alkali reagent for MW pretreatment and enzymatic saccharification of sweet sorghum bagasse, whereas sodium and ammonium hydroxides were excellent with MW pretreatment and enzymatic saccharification in high yields of sugars depending on the biomass used.

Figure 2 shows the various sugar analysis methods applied in the last decade in quantifying the sugar yields from MW-assisted alkali pretreatment and enzymatic hydrolysis process of cellulosic biomass. The most widely applied method is DNS with 67% followed by NREL protocol with 28% over the last 10 years as indicated in the published research papers. None of the research studies that have used these methods indicated the most appropriate method. Rather, results of sugar yields were based on the type of biomass used and pretreatment parameters. However, there was no analysis on the cost of using any of the sugar analysis methods reported in the study.

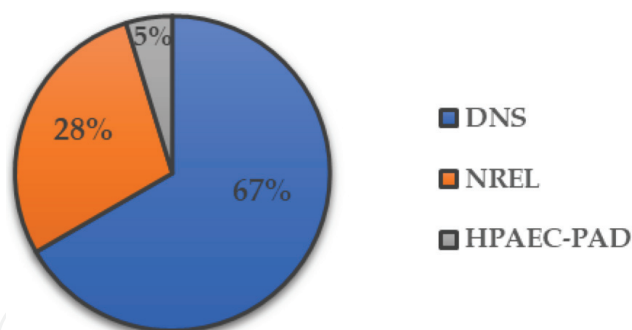


Figure 2. Cellulosic sugar analysis methods.

On the other hand, an overview of key pretreatment processes employed for the bioconversion of lignocellulosic biomass was reported by Chaturvedi and Verma [79]. The study suggested that alkali pretreatment process involving lime, ammonia, NaOH, and KOH resulted in higher yields of sugars involving lignocellulosic biomass with a low lignin content like rice hull and grasses. But concerns with environment challenges were associated with ammonia because it is toxic to the environment.

2.4. Effect of biomass pelleting on enzymatic digestibility

Densification of biomass is primarily achieved by pelletizing which is the application of mechanical force to compact biomass into uniformly sized solid particles [80, 81]. Densification increases the density of biomass into a pellet product having a density of 600–1200 kg/m³ [82] for efficient transportation and low moisture for safe storage [83]. Particle size and preconditioning of biomass prior to pelletization can facilitate the binding characteristics and chemical composition of biomass, thereby improving the overall pellet quality [84]. In addition, moisture content as a factor during preheating of biomass before pelleting assists in loosening the natural binders to produce durable pellets [85].

The pretreatment process helps to complete biomass conversion into valuable bioproducts. Therefore, the pretreatment of lignocellulosic biomass is important in enhancing enzymatic cellulosic digestibility to increase glucose yields [86]. There is only one cited paper on the effects of MW-assisted alkali pretreatment and densification on improving enzymatic saccharification of biomass conversion into ethanol. Sugar yields were reported to increase after MW-assisted alkali pretreatments of canola straw and oat hull pellets. Sodium hydroxide (NaOH) and potassium hydroxide (KOH) at various concentrations were used in the study. The authors highlighted that samples selected for cellulosic substrate analysis were based on parameters that describe pellet quality such as tensile strength, dimensional stability, and pellet density [3].

Table 2 shows MW-assisted alkali canola straw and oat hull pellet data and corresponding glucose yield results. The tensile strength, dimensional stability, and pellet density showed little or no significant effect on the sugar yields on canola straw and oat hull pellets. It is evident that samples ground in a 1.6-mm hammer mill screen size had a significant effect on the cellulosic enzymatic digestibility. **Table 2** shows data and results from Hoover et al. [85] and Shi et al. [87] which were compared. Hoover et al. [85] indicated that preheating AFEX-pretreated biomass pellets had no effect on sugar yield while the non-preheated pellet had a

Feedstock	Screen size (mm)	Alkali	Alkali concentration (%)	MW time (min)	Tensile strength (MPa)	Dimensional stability (%)	Pellet density (kg/m ³)	Average glucose yield (mg/g)
Canola straw	1.6	NaOH	1.5	18	2.31	0.79	1370.27	110.05
		KOH	1.5	6	3.78	0.83	1392.21	53.42
	3.2	NaOH	0.75	6	4.85	2.60	1324.75	55.78
		KOH	1.5	6	5.19	0.83	1382.62	96.77
Oat hull	1.6	NaOH	0.75	18	1.33	5.28	1221.99	99.10
		KOH	1.5	6	0.63	1.04	1185.69	97.53
	3.2	NaOH	0.75	6	1.23	1.68	1205.73	72.22
		KOH	1.5	18	1.17	5.95	1210.94	84.87

Table 2. MW-assisted alkali pretreated canola straw and oat hull pellets and glucose yields results [3].

greater effect on the sugar yield. Also, Shi et al. [87] reported that efficiency in the mixed feedstocks pretreatments and densification demonstrated significant effect on sugar yields. Many studies without MW-assisted alkali pretreatment method using pelletization on different biomass have reported similar sugar yield, considering the biomass used in the conversion. Furthermore, conclusions made from these studies focused more on the effects of pelletization parameters on improving enzymatic hydrolysis process for biomass sugar conversion [85, 87–91]. The advantages of densifying biomass using different technologies to produce pellets are to improve handling, storage, and transportation efficiencies [92].

2.5. Economic evaluation of microwave pretreatment process

Technoeconomic analysis (TEA) involves technologies, system, and production processes evaluation. Different technoeconomic studies have classified the analysis into two major groups such as technical (maintenance requirement and service life, operation and maintenance skill requirement, the ease of transportation and installation, processing capacities available, material, esthetic and inherent risk for a system or product process) and economic (capital and operating costs, biomass cost, and profit revenues) depending on the process technology used [93, 94]. The TEA report also assists in understanding and providing additional information to the economic viability via production cost and market price [94], and the profitability and sensitivity analysis of a product or a process [95, 96]. To perform technical and economic evaluation, various software with in-built analysis tool to estimate capital and operational costs have been developed by different software developers, and the choice of software is dependent on the project evaluation. The available commercial TEA software includes Super-Pro designer, PRO/II and DYNsIM, Aspen Plus HYSYS, DESIGN II, and CHEMCAD [93].

Life cycle assessment (LCA) involves the collection and evaluation of relevant input and output data of a product system including potential environment impacts within the process period [97]. Adams et al. [98] indicated that the main reason for using the LCA tool is to give a

holistic view of the emissions and resource requirements of a product system. Also, the importance of LCA study is to analyze the impact on the environment, energy consumption, and economic viability [94, 99]. Azapagic and Stichnothe [100] reported that LCA can be translated into quantitative measures of sustainability such as environmental, social, and economic. Different types of International Standard Organization (ISO) documents have been developed for LCA standards in providing flexible methodology and enabling modification of analysis by meeting up the goal and scope of the study [94, 97]. In the setting of innovation targets, the major impacts of LCA can be identified using these intensive products: raw material, manufacturing, distribution, the use of intensive product such as automobiles and laser printers, and disposal-intensive product [94, 100]. Many software, tools, and databases have been developed to assist in data processing and calculation of LCAs. These include Athena, BEES 4.0, CMLCA, Ecolnvent, EMIS, GaBi, GEMIS, IdeMAT, REGIS, SimaPro, and Umberto [101]. Many software are in market and many more are disappearing each year due to the dynamic nature and availability of the software [94]. Patel et al. [102] studied the technoeconomic and life cycle assessment of lignocellulosic biomass thermochemical conversion technologies. The study stated that a lot of research works are only focusing attention on calculating the cost of one specific production chain product. It suggested more technoeconomic investigation on multiple processes of product co-generation. Mupondwa et al. [103] reported that bioenergy research investigations and developments in Canada for biomass pretreatments and conversions into bioproducts using different technologies have recently shown significant growth. However, the study highlighted that there are challenges in designing business models and commercial bioconversion pathways based on various biomass feedstocks available. Despite the varieties of research that have been conducted on microwave pretreatment technique, the growth of industrial application of microwave heating is limited globally. Xu [31] reported that most pretreatment experiments still take place in domestic MW ovens except for few modified MW reactors which are used by less than 30 companies globally on the pretreatment of various feedstocks. The study on the TEA of MW-assisted alkali pretreatment is not available except for TEA on MW torrefaction and pyrolysis of biomass. Even with TEA on MW torrefaction or pyrolysis published papers displayed, an extensive literature search still showed limited information on the economic evaluation on the processes. Therefore, the technoeconomics of MW-assisted alkali pretreatment of biomass needs to be evaluated using available data to determine economic viability of the process, and this will be one of the topics of research that our group will undertake.

3. Summary

MW pretreatment technique has gained research attention and its future is growing. In spite of this, it is still under bench-scale development. Sufficient data generated from previous and recent studies can be used to quantify the dielectric properties of input biomass and to design and develop a continuous MW-assisted pretreatment and enzymatic saccharification process unit for commercial scale-up. Feedstock properties and reaction conditions are the two factors influencing microwave pretreatment characterization and yield of the final product.

The cost of MW pretreatment process is the main determinant associated with the adoption of this technology. Emphasis on the capital and operating cost investment will improve the pretreatment technique and hydrolysis process, thereby reducing the cost of the final product (bioethanol). Future research studies in MW-assisted biological pretreatment in improving lignocellulosic biomass digestibility, and techno-economic analysis and life cycle impact assessment of the process need to be considered. The concept is to develop a more economic pretreatment and hydrolysis techniques that can be environmentally sustainable and accepted by the bioenergy industry.

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Conflict of interest

The authors declare no conflict of interest.

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References

- [1] Smith D. Brief overview of biofuels. Report 2 and 3 [Internet]. McGill University, Montreal, QC: Biofuelnet Canada. 2013. Available from: <http://biofuelnet.ca/advanced-biofuels-course> [Accessed: April 11, 2017]

- [2] Iroba KL, Tabil LG, Sokhansanj S, Meda V. Producing durable pellets from barley straw subjected to radio frequency-alkaline and steam explosion pretreatments. *International Journal of Agricultural and Biological Engineering*. 2014;**7**(3):68-82. DOI: 10.3965/j.ijabe.20140703.009
- [3] Agu OS, Tabil LG, Dumonceaux T. Microwave-assisted alkali pretreatment, densification and enzymatic saccharification of canola straw and oat hull. *Bioengineering*. 2017;**4**(25): 1-33. DOI: 10.3390/bioengineering4020025
- [4] Yin C. Microwave-assisted pyrolysis of biomass for liquid biofuels production. *Journal of Bioresource Technology*. 2012;**120**:273-284. DOI: 10.1016/j.biortech.2012.06.016
- [5] Bhaskar T, Bhavya B, Singh R, Naik DV, Kumar A, Goyal HB. Thermochemical conversion of biomass to biofuels. In: Pandey A, Gnansounou E, Dussap CG, Larroche C, Ricke SC, editors. *Biofuels: Alternative Feedstocks and Conversion Processes*. Oxford, UK: Academic Press; 2011. pp. 51-77
- [6] Aho A, Salmi T, Murzin DY. Catalytic pyrolysis of lignocellulosic biomass. In: Triantafyllidis KS, Lappas AA, Stocker M, editors. *The Role of Catalyst for the Sustainable Production of Biofuels and Bio-Chemicals*. Netherlands: Elsevier; 2013. pp. 137-145. DOI: 10.1016/B978-0-444-56330-9.00005-X
- [7] Tong Z, Pullammanappallil P, Teixeira AA. How ethanol is made from cellulosic biomass [Internet]. 2012. Available from: <http://edis.ifas.ufl.edu/pdffiles/AE/AE49300.pdf> [Accessed: March 17, 2018]
- [8] Karimi K, Shafiei M, Kumar R. Progress in physical and chemical pretreatment of lignocellulosic biomass. In: Gupta VK, Tuohy MG, editors. *Biofuels Technologies*. Berlin, Germany: Springer; 2013. pp. 53-96
- [9] Sarkar N, Ghosh SK, Bannerjee S, Aikat K. Bioethanol production from agricultural waste: An overview. *Renewable Energy*. 2012;**37**:19-27. DOI: 10.1016/j.renene.2011.06.045
- [10] Kim S, Dale BE. Environmental aspects of ethanol derived from no-tilled corn grain: Non-renewable energy consumption and greenhouse gas emission. *Biomass and Bioenergy*. 2005;**28**:475-489. DOI: 10.1016/j.biombioe.2004.11.005
- [11] Nomanbhay SM, Hussain R, Palanisamy K. Microwave-assisted alkaline pretreatment and microwave assisted enzymatic saccharification of oil palm empty fruit bunch fiber for enhance fermentable sugar yield. *Journal of Sustainable Bioenergy System*. 2013;**3**:7-17. DOI: 10.4236/jsbs.2013.31002
- [12] Quintero JA, Rincon LE, Cardona CA. Production of bioethanol from agro industrial residues as feedstocks. In: Pandey A, Gnansounou E, Dussap CG, Larroche C, Ricke SC, editors. *Biofuels: Alternative Feedstocks and Conversion Processes*. Oxford, UK: Academic Press; 2011. pp. 253-260
- [13] Van Zessen E, Weismann M, Bakker RRC, Elbersen HW, Reith JH, den Uil H. Lignocellulosic ethanol, a second opinion [Internet]. 2003. Available from: <http://library.wur.nl/WebQuery/wurpubs/339809> [2018/01/28]

- [14] Harmsen PFH, Huijgen WJJ, Lopez LMB, Bakker R. Literature Review of Physical and Chemical Pretreatment Processes for Lignocellulosic Biomass. Energy research center of the Netherland. 2010;**10**(13):1-54
- [15] Mosier NS, Wyman C, Dale B, Elander R, Lee YY, Holtzapple M, Ladisch MR. Features of promising technologies for pretreatment of lignocellulosic biomass. Bioresource Technology. 2005;**96**(6):673-686. DOI: 10.1016/j.biortech.2004.06.025
- [16] Agbor VB, Cicek N, Sparling R, Berlin A, Levin BD. Biomass pretreatment: Fundamentals toward application. Biotechnology Advances. 2011;**29**. DOI: 675-685. DOI: 10.1016/j.biotechadv.2011.05.005
- [17] Energy Efficiency and Renewable Energy U.S. Department of Energy Biomass Program [Internet]. 2012. Available from: https://www.energy.gov/sites/prod/files/2014/03/f14/obp_overview_algae_summit.pdf (2018/03/27)
- [18] Aguilar-Reynosa A, Romani A, Rodriguez-Jasso RM, Aguilar CN, Garrote G, Ruiz HA. Microwave heating processing as alternative of pretreatment in second-generation biorefinery: An overview. Energy Conversion and Management. 2017;**136**:50-65. DOI: 10.1016/j.enconman.2017.01.004
- [19] Agu OS. Microwave-assisted alkali pretreatment, densification and enzymatic saccharification of canola straw and oat hull [thesis]. Saskatoon: University of Saskatchewan; 2017
- [20] Alvira P, Tomas-Pejo E, Ballesteros M, Negro MJ. Pretreatment technologies for an efficient bioethanol production process based on enzymatic hydrolysis: A review. Bioresource Technology. 2010;**101**:4851-4861. DOI: 10.1016/j.biortech.2009.11.093
- [21] Kumar P, Barrett DM, Delwiche MJ, Stroeve P. Methods for pretreatment of lignocellulosic biomass for efficient hydrolysis and biofuel production. Industrial and Engineering Chemistry Research. 2009;**48**:3713-3729. DOI: 10.1021/ie801542g
- [22] Merino-Perez O, Martinez-Palou R, Labidi J, Luque R. Microwave-assisted pretreatment of lignocellulosic biomass to produce biofuels and value-added products. In: Fang Z, Smith RL Jr, Qi X, editors. Production of Biofuels and Chemicals with Microwave, Biofuels and Biorefineries. Dordrecht, Netherlands: Springer; 2015. pp. 197-223. DOI: 10.1007/978-94-017-9612-5_10
- [23] Rosgaard L, Peddersen S, Meyer AS. Comparison of different pretreatment strategies for enzymatic hydrolysis of wheat and barley straw. Applied Biochemistry and Biotechnology. 2007;**143**:284-296. DOI: 10.1007/s12010-007-8001-6
- [24] Silverstein RA, Chen Y, Sharma-Shivappa RR, Boyette MD, Osborne JA. Comparison of chemical pretreatment methods for improving saccharification of cotton stalks. Bioresource Technology. 2007;**98**:3000-3011. DOI: 10.1016/j.biortech.2006.10.022
- [25] Chen C, Boldor D, Aita G, Walker M. Ethanol production from sorghum by a microwave-assisted dilute ammonia pretreatment. Bioresource Technology. 2012;**110**:190-197. DOI: 10.1016/j.biortech.2012.01.021

- [26] Chiaramonti D, Prussi M, Ferrero S, Oriani L, Ottonello P, Torre P, Cherchi F. Review of pretreatment processes for lignocellulosic ethanol production, and development of an innovative method. *Biomass and Bioenergy*. 2012;**46**:25-35. DOI: 10.1016/j.biombioe.2012.04.020
- [27] Rodríguez-Jasso RM, Mussatto SI, Pastrana L, Aguilar CN, Teixeira JA. Microwave-assisted extraction of sulfated polysaccharides (fucoidan) from brown seaweed. *Carbohydrate Polymers*. 2011;**86**(3):1137-1144. DOI: 10.1016/j.carbpol.2011.06.006
- [28] Decareau RV, Peterson RA. *Microwave Processing and Engineering*. USA: Wiley-VCH Verlag; 1986. p. 1986, 224
- [29] Kappe CO, Stadler A, Dallinger D, Mannhold R, Kubinyi H, Folkers G. In: Mannhold R, Kubinyi H, Folkers G, editors. *Microwaves in Organic and Medicinal Chemistry*. 2nd completely revised and enlarged ed. USA: John Wiley and Sons; 2012. p. 683
- [30] Meda VV, Orsat VR. Microwave heating and the dielectric properties of foods. In: *Microwave Process Foods*. 2nd ed. Duxford, UK: Woodhead; 2017. pp. 23-43. DOI: 10.1016/B978-0-08-100528-6.00020-6
- [31] Xu J. Microwave pretreatment. In: Pandey A, editor. *Pretreatment of Biomass: Processes and Technologies*. Waltham, MA, USA: Elsevier; 2015. pp. 157-172
- [32] Motasemi F, Afzal MT. A review on the microwave-assisted pyrolysis technique. *Renewable and Sustainable Energy Reviews*. 2013;**28**:317-330. DOI: 10.1016/j.rser.2013.08.008
- [33] Tsubaki S, Azuma J. Application of microwave Technology for Utilization of recalcitrant biomass. In: Grundas SA, editor. *Advances in Induction and Microwave Heat Mineral Organic Materials*. Croatia, Rijeka: InTech; 2011. pp. 1-27. DOI: 10.5772/14040
- [34] Datta AK. Fundamentals of heat and moisture transport for microwaveable food product and process development. In: Datta AK, Anantheswaran RC, editors. *Handbook of Microwave Technology for Food Application*. New York: M. Dekker; 2001. p. 536
- [35] Sun J, Wang W, Yue Q, Ma C, Zhang J, Zhao X, Song Z. Review on microwave-metal discharges and their applications in energy and industrial processes. *Applied Energy*. 2016;**175**:141-157. DOI: 10.1016/j.apenergy.2016.04.091
- [36] Horikoshi S, Osawa A, Sakamoto S, Serpone N. Control of microwave generated hot spots. Part IV. Control of hot spots on a heterogeneous microwave-absorber catalyst surface by a hybrid internal/external heating method. *Chemical Engineering and Processing: Process Intensification*. 2013;**69**:52-56. DOI: 10.1016/j.cep.2013.02.003
- [37] Campañone LA, Paola CA, Mascheroni RH. Modeling and simulation of microwave heating of foods under different process schedules. *Food and Bioprocess Technology*. 2010;**5**:738-749. DOI: 10.1007/s11947-010-0378-5
- [38] Taherzadeh MJ, Karimi K. Pretreatment of lignocellulosic waste to improve ethanol and biogas production: A review. *International Journal of Molecular Sciences*. 2008;**9**(9):1621-1651. DOI: 10.3390/ijms9091621

- [39] Sindhu R, Pandey A, Binod P. Alkaline treatment. In: Pandey A, Negi S, Binod P, Larroche C, editors. *Pretreatment of Biomass: Process and Technologies*. Oxford, UK: Elsevier; 2015. pp. 51-60
- [40] Karunanithy C, Muthukumarappan K. Optimization of alkali, big bluestem particle size and extruder parameters for maximum enzymatic sugar recovery using response surface. *BioResources*. 2011;6(1):762-790. DOI: 10.15376/biores.6.1.762-790
- [41] Ooshima H, Aso K, Harano Y, Yamamoto T. Microwave treatment of cellulosic materials for their enzymatic hydrolysis. *Biotechnology Letters*. 1984;6(5):289-294. DOI: 10.1007/BF00129056
- [42] Azuma JI, Tanaka F, Koshijima T. Enhancement of enzymatic susceptibility of lignocellulosic wastes by microwave irradiation. *Journal of Fermentation Technology*. 1984;62(4):377-384
- [43] Gong G, Liu D, Huang Y. Microwave-assisted organic acid pretreatment for enzymatic hydrolysis of rice straw. *Biosystems Engineering*. 2010;107:67-73. DOI: 10.1016/j.biosystemseng.2010.05.012
- [44] Keshwani DR, Cheng JJ. Microwave-based alkali pretreatment of switchgrass and coastal bermudagrass for bioethanol production. *Biotechnology Progress*. 2010;26(3):644-652. DOI: 10.1002/btpr.371
- [45] Quitain AT, Sasaki M, Goto M. Microwave-based pretreatment for efficient biomass-to-biofuel conversion. In: Fang Z, editor. *Pretreatment Techniques for Biofuels and Biorefineries*. Berlin, Germany: Springer; 2013. pp. 117-130. DOI: 10.1007/978-3-642-32735-3_6
- [46] Islam MR, Gupta A, Rivi M, Beg MDH. Characterization of microwave-treated oil palm empty fruit bunch/glass fibre/polypropylene composites. *Journal of Thermoplastic Composite Materials*. 2015:1-17. DOI: 10.1177/0892705715614078
- [47] Ethaib S, Omar R, Kamal SMM, Biak DRA. Microwave-assisted pretreatment of lignocellulosic biomass: A review. *Journal of Engineering Science and Technology*. 2015;2(1):97-109
- [48] Tomas-Pejo E, Alvria P, Ballesteros M, Negro MJ. Pretreatment technologies for lignocellulose to bioethanol conversion. In: Pandey A, Gnansounou E, Dussap CG, Larroche C, Ricke SC, editors. *Biofuels: Alternative Feedstocks and Conversion Processes*. Oxford, UK: Academic Press; 2011. pp. 149-177
- [49] Darji D, Alias Y, Som FMRN. Microwave heating and hydrolysis of rubber wood biomass in ionic liquids. *Journal of Chemical Technology & Biotechnology*. 2015;90:2050-2056. DOI: 10.1002/jctb.4516
- [50] Stickel JJ, Elander RT, McMillan JD, Brunecky R. Enzymatic hydrolysis of lignocellulosic biomass. In: Bisaria SV, Kondo A, editors. *Bioprocessing of Renewable Resources to Commodity Biproducts*. 1st ed. New York, NY: John Wiley and Sons; 2014. pp. 77-103. DOI: 10.1002/9781118845394.ch4

- [51] Geddes CC, Peterson JJ, Roslander C, Zacchi G, Mullinnix MT, Shanmugam KT, Ingram LO. Optimizing saccharification of sugar cane bagasse using dilute phosphoric acid followed by fungal cellulases. *Bioresource Technology*. 2010;**101**:1851-1857. DOI: 10.1016/j.biortech.2009.09.070
- [52] Maitan-Alfenas GP, Visser EM, Guimaraes VM. Enzymatic hydrolysis of lignocellulosic biomass: Converting food waste in valuable products. *Current Opinion in Food Science*. 2015;**1**:44-49. DOI: 10.1016/j.cofs.2014.10.001
- [53] Lan TQ, Lou H, Zhu JY. Enzymatic saccharification of lignocelluloses should be conducted at elevated pH 5.2–6.2. *Bioenergy Research*. 2013;**6**(2):476-485. DOI: 10.1007/s12155-012-9273-4
- [54] Brummer V, Skryja P, Jurena T, Hlavacek V, Stehlik P. Suitable technological conditions for enzymatic hydrolysis of waste paper by Novozymes(R) enzymes NS50010. *Applied Biochemistry and Biotechnology*. 2014;**174**:1299-1308. DOI: 10.1007/s12010-014-1119-4
- [55] Binod P, Janu KU, Sindhu R, Pandey A. Hydrolysis of lignocellulosic biomass for bioethanol production. In: Pandey A, Gnansounou E, Dussap CG, Larroche C, Ricke SC, editors. *Biofuels: Alternative Feedstocks and Conversion Processes*. Oxford, UK: Academic Press; 2011. pp. 229-250
- [56] Van Dyk JS, Pletschke BI. A review of lignocellulose bioconversion using enzymatic hydrolysis and synergistic cooperation between enzymes – Factors affecting enzymes, conversion and synergy. *Biotechnology Advances*. 2012;**30**(6):1458-1480. DOI: 10.1016/j.biotechadv.2012.03.002
- [57] Kumar D, Murthy GS. Stochastic molecular model of enzymatic hydrolysis of cellulose for ethanol production. *Biotechnology for Biofuels*. 2013;**6**(63):1-20. DOI: 10.1186/1754-6834-6-63
- [58] Amore A, Glacobbè S, Faraco V. Regulation of cellulase and hemicellulase gene expression in fungi. *Current Genomics*. 2013;**14**:230-249. DOI: 10.2174/1398202911314040002
- [59] de Almeida MN, Falkoski DL, Guimaraes VM, Ramos HJ, Visser EM, Maitan-Alfenas GP, de Rezende ST. Characteristics of free endoglucanase and glycosidases multienzyme complex from *Fusarium verticillioides*. *Bioresource Technology*. 2013;**143**:413-422. DOI: 10.1016/j.biortech.2013.06.021
- [60] Chang V, Holtzapple MT. Fundamental factors affecting biomass enzymatic reactivity. *Applied Biochemistry and Biotechnology*. 2000;**1**:84, 5-86, 37. DOI: 10.1007-978-1-4612-1392-5_1
- [61] Laureano-Perez L, Teymouri F, Alizadeh H, Dale BE. Understanding factors that limit enzymatic hydrolysis of biomass. *Applied Biochemistry and Biotechnology*. 2005;**121–124**:1081-1099. DOI: 10.1007/978-1-59259-991-2_91
- [62] Palonen H, Bjerre AB, Tenkanen M, Viikari L. Evaluation of wet oxidation pretreatment for enzymatic hydrolysis of softwood. *Applied Biochemistry and Biotechnology*. 2004;**117**(1):1-17. DOI: 10.1385/ABAB:117:1:01

- [63] Janker-Obermeier I, Sieber V, Faulstich VM, Schieder D. Solubilization of hemicellulose and lignin from wheat straw through microwave-assisted alkali treatment. *Industrial Crops and Products*. 2012;**39**:198-203. DOI: 10.1016/j.indcrop.2012.02.022
- [64] Sun Y, Cheng J. Hydrolysis of lignocellulosic materials for ethanol production: A review. *Bioresource Technology*. 2002;**83**(1):1-11. DOI: 10.1016/S0960-8524(01)00212-7
- [65] Zhu S, Wu Y, Yu Z, Zhang X, Wang C, Yu F, Jin S. Production of ethanol from microwave-assisted alkali pretreated wheat straw. *Process Biochemistry*. 2006;**41**(4):869-873. DOI: 10.1016/j.procbio.2005.10.024
- [66] Hu ZH, Wen ZY. Enhancing enzymatic digestibility of switchgrass by microwave assisted alkali pretreatment. *Biochemical Engineering Journal*. 2008;**38**:369-378. DOI: 10.1016/j.bej.2007.08.001
- [67] Keshwani DR, Cheng JJ, Burns JC, Li L, Chiang V. Microwave pretreatment of switchgrass to enhance enzymatic hydrolysis. In: *Proceedings of the ASABE Annual International Meeting*; 17–20 June 2007; Minneapolis, Minnesota. St. Joseph, MI: ASABE; 2007. Paper No. 077127. pp. 1-8
- [68] Corredor DY. Pretreatment and enzymatic hydrolysis of lignocellulosic biomass [thesis]. Manhattan, Kansas: Department of Biological and Agricultural Engineering, Kansas State University; 2008
- [69] Hamzah F, Idris A, Rashid R, Ming SJ. Lactic acid production from microwave alkali pretreated empty fruit bunches fibre using *Rhizopus oryzae* pellet. *Journal of Applied Science*. 2009;**9**:3086-3091. DOI: 10.3923/jas.2009.3086.3091
- [70] Wang LL, Han G, Zhang Y. Comparative study of composition, structure and properties of *Apocynum venetum* fibers under different pretreatments. *Carbohydrate Polymers*. 2007;**69**(2):391-397. DOI: 10.1016/j.carbpol.2006.12.028
- [71] Diaz AB, Moretti MM, Bezerra-Bussoli C, C Nunes Cda C, Blandino A, da Silva R, Gomes E. Evaluation of microwave-assisted pretreatment of lignocellulosic biomass immersed in alkaline glycerol for fermentable sugars. *Bioresource Technology*. 2015;**185**:316-323. DOI: 10.1016/j.biortech.2015.02.112
- [72] Binod P, Satyanagalakshmi K, Sindhu R, Janu KU, Sukumaran RK, Pandey A. Short duration microwave assisted pretreatment enhances the enzymatic saccharification and fermentable sugar yield from sugarcane bagasse. *Renewable Energy*. 2012;**37**:109-116. DOI: 10.1016/j.renene.2011.06.007
- [73] Choudhary R, Umagiliyage AL, Liang Y, Siddaramu T, Haddock J, Markevicius G. Microwave pretreatment for enzymatic saccharification of sweet sorghum bagasse. *Biomass and Bioenergy*. 2012;**39**:218-226. DOI: 10.1016/j.biombioe.2012.01.006
- [74] Xu J, Chen H, Kadar Z, Thomsen AB, Schmidt JE, Peng H. Optimization of microwave pretreatment on wheat straw for ethanol production. *Biomass and Bioenergy*. 2011;**35**(9): 3859-3864. DOI: 10.1016/j.biombioe.2011.04.054

- [75] Singh R, Tiwari S, Srivastava M, Shukla A. Microwave assisted pretreatment of rice straw for enhancing enzymatic digestibility. *Journal of Energy*. 2014;**2014**:1-7. DOI: 10.1155/2014/03483813
- [76] Conesa CL, Segui N, Laguarda-Miro N, Fito P. Microwave-assisted alkali pretreatment for enhancing pineapple waste saccharification. *BioResources*. 2016;**11**(3):6518-36531. DOI: 10.1537/biores.11.3.6518-6531
- [77] Rodrigues THS, Rocha MVP, De MacEdo GR, Goncalves LRB. Ethanol production from cashew apple bagasse: Improvement of enzymatic hydrolysis by microwave-assisted alkali pretreatment. *Applied Biochemistry and Biotechnology*. 2011;**164**:929-943. DOI: 10.1007/s12010-011-9185-3
- [78] Jin S, Zhang G, Zhang P, Li F, Wang S, Fan S, Zhou S. Microwave assisted alkaline pretreatment to enhance enzymatic saccharification of catalpa sawdust. *Bioresource Technology*. 2016;**221**:26-30. DOI: 10.1016/j.biortech.2016.09.033
- [79] Chaturvedi V, Verma P. An overview of key pretreatment processes employed for bio-conversion of lignocellulosic biomass into biofuels and value-added products. *Biotechnology*. 2013;**3**:415-443. DOI: 10.1007/s13205-013-0167-8
- [80] Veal MW. Biomass logistics. In: Cheng J, editor. *Biomass to Renewable Energy Processes*. Boca Raton, FL, USA: CRC Press; 2010. pp. 71-130
- [81] Chen WH, Peng J, Bi XT. A state-of-the-art review of biomass torrefaction, densification and applications. *Renewable and Sustainable Energy Reviews*. 2015;**44**:847-866. DOI: 10.1016/j.rser.2014.12.039
- [82] Kashaninejad M, Tabil LG. Effect of microwave: Chemical pretreatment on compression characteristics of biomass grinds. *Biosystems Engineering*. 2011;**108**(1):36-45. DOI: 10.1016/j.biosystemseng.2010.10.008
- [83] Mani S, Tabil LG, Sokhansanj S. Effects of compressive force, particle size and moisture content on mechanical properties of biomass pellets from grasses. *Biomass & Bioenergy*. 2006;**30**(7):648-654. DOI: 10.1016/j.biombioe.2005.01.004
- [84] Tumuluru JS, Tabil LG, Opoku A, Mosqueda MR, Fadeyi O. Effects of process variables on the quality characteristics of pelleted wheat distiller's dried grains with solubles. *Biosystems Engineering*. 2010;**105**(4):466-475. DOI: 10.1016/j.biosystemseng.2010.01.005
- [85] Hoover AN, Tumuluru JS, Teymouri F, Moore J, Gresham G. Effect of pelleting process variables on physical properties and sugar yields of ammonia fiber expansion pretreated corn Stover. *Bioresource Technology*. 2014;**164**:128-135. DOI: 10.1016/j.biortech.2014.02.005
- [86] Himmel ME, Ding SY, Johnson DK, Adney WS, Nimlos MR, Brady JW, Foust TD. Biomass recalcitrance: Engineering plants and enzyme for biofuels production. *Science*. 2007;**315**:804-807. DOI: 10.1126/science.1137016

- [87] Shi J, Thompson VS, Yancey NA, Stavila V, Simmons BA, Singh S. Impact of mixed feedstocks and feedstock densification on ionic liquid pretreatment efficiency. *Biofuels*. 2013;**4**(1):63-72. DOI: 10.4155/bfs.12.82
- [88] Bals BD, Gunawan C, Moore J, Teymouri F, Dale BE. Enzymatic hydrolysis of pelletized AFEX treated corn Stover at high solid loadings. *Biotechnology and Bioengineering*. 2013;**111**(2):264-271. DOI: 10.1002/bit.25022
- [89] Chundawat SPS, Donohoe BS, da C, Sousa L, Elder T, Agarwal UP, Lu F, Ralph J, Himmel ME, Balan V, Dale BE. Multi-scale visualization and characterization of lignocellulosic plant cell wall deconstruction during thermochemical pretreatment. *Energy and Environmental Science*. 2011;**4**. DOI: 973 – 984. DOI: 10.1039/c0ee00574f
- [90] Rijal B, Igathinathane C, Karki B, Yu M, Pryor SW. Combined effect of pelleting and pretreatment on enzymatic hydrolysis of switchgrass. *Bioresource Technology*. 2012;**116**: 36-41. DOI: 10.1016/j.biortech.2012.04.054
- [91] Theerarattananon K, Xu F, Wilson J, Staggenborg S, McKinney L, Vadlani P, Pei Z, Wang D. Effects of the pelleting conditions on chemical composition and sugar yield of corn Stover, big bluestem, wheat straw, and sorghum stalk pellets. *Bioprocess and Biosystems Engineering*. 2012;**35**(4):615-623. DOI: 10.1007/s00449-011-0642-8
- [92] Adapa PK, Tabil LG, Schoenau G. Factors affecting the quality of biomass pellet for biofuel and energy analysis of pelleting process. *International Journal of Agricultural and Biological Engineering*. 2013;**6**(2):1-12. DOI: 10.3965/j.ijabe.20130602.001
- [93] Towler G, Sinnott R. *Chemical Engineering Design, Principles, Practice and Economics of Plant and Process Design*. 2nd ed. New York: Elsevier Ltd; 2012. p. 1320
- [94] Shah A, Baral NR, Manandhar A. Technoeconomic analysis and life cycle assessment of bioenergy systems. In: *Advance in Bioenergy*. 2016;**1**:189-247. DOI: 10.1016/bs.aibe.2016.09.004
- [95] Li X, Mupondwa E, Tabil LG. Technoeconomic analysis of biojet fuel production from camelina at commercial scale: Case of Canadian prairies. *Bioresource Technology*. 2018;**249**:196-205. DOI: 10.1016/j.biortech.2017.09.183
- [96] Mupondwa E, Li X, Tabil LG, Adapa P, Sokhansanj S, Stumborg M, Gruber M, Laberge S. Technoeconomic analysis of wheat straw densification in the Canadian prairie province of Manitoba. *Bioresource Technology*. 2012;**110**:355-363. DOI: 10.1016/j.biortech.2012.01.100
- [97] International Organization Standard. *Environmental Management – Life Cycle Assessment – Principles and Framework/Requirements and Guidelines*. Geneva, Switzerland; 2006. 14040/44
- [98] Adams PWR, Shirley JEJ, McManus MC. Comparative cradle-to-gate life cycle assessment of wood pellet production with torrefaction. *Applied Energy*. 2015;**138**:367-380. DOI: 10.1016/j.apenergy.2014.11.002

- [99] Roy P. Life cycle assessment of ethanol produced from lignocellulosic biomass: techno-economic and environmental evaluation [thesis]. Guelph Ontario: University of Guelph; 2014
- [100] Azapagic A, Stichnothe H. Sustainability assessment of biofuels. In: Azapagic A, Perdan S, editors. Sustainable Development in Practice: Case Studies. John Wiley and Sons; 2010. pp. 142-169. DOI: 10.1002/9780470972847
- [101] Ciroth A. Software for life cycle assessment. In: Curran MA, editor. Life Cycle Assessment Handbook: A Guide for Environmental Sustainable Products. Salem, MA: Scrivener Publishing LLC; 2012. DOI: 10.1002/9781118528372.ch6
- [102] Patel M, Zhang X, Kumar A. Technoeconomic and life cycle assessment on lignocellulosic biomass thermochemical conversion technologies: A review. Renewable and Sustainable Energy Reviews. 2016;**53**:1486-1499. DOI: 10.1016/j.rser.2015.09.070
- [103] Mupondwa E, Xue L, Tabil LG, Sokhansanj S, Adapa P. Status on Canada's lignocellulosic ethanol: Part I: Pretreatment technologies. Renewable and Sustainable Energy Reviews. 2017;**72**:178-190. DOI: 10.1016/j.rser.2017.01.039

