Moderate pretreatment strategies for improvement of reducing sugar production from oil palm empty fruit bunches

B M Harahap¹, M R Maulid¹, A I Dewantoro¹, E Mardawati¹, and S Huda²

¹Department of Agroindustrial Technology, Faculty of Agroindustrial Technology, Universitas Padjadjaran, Indonesia
²Department of Food Industrial Technology, Faculty of Agroindustrial Technology, Universitas Padjadjaran, Indonesia

E-mail: budi.mandra.harahap@unpad.ac.id

Abstract. Pretreatment at mild condition is the strategy to minimize energy consumption, to reduce toxic compounds influencing to further process, and to obtain the high reducing sugar yield as fermentation substrate from cellulose and hemicellulose of oil palm empty fruit bunches (OPEFB). The yield still could be increased by optimizing pretreatment condition and varying several process alternatives. In this study, the effect of acetic acid addition and its corresponding concentration (5–15%), pretreatment temperature (115–125 °C), time (60–90 minutes), and two-stage pretreatment (0.01–1M NaOH pretreatment followed by autohydrolysis and autohydrolysis followed by 0.1–1% acetic acid pretreatment) were evaluated. The residual solid after pretreatment was then enzymatically hydrolyzed by Cellic CTec2. The result showed that the addition of acetic acid was able to increase the yield of reducing sugar. In addition, the reducing sugar yield could be still increased when the temperature was escalated, but the extended pretreatment time gave the decline of reducing sugar yield. This yield, however, was still lower than this from NaOH pretreatment followed by autohydrolysis. The maximum yield of this pretreatment could be attained by 1 M NaOH concentration at 125 °C for 60 minutes with solid loading 10% (0.71 g/g-OPEFB).

Keywords OPEFB, Reducing Sugar, Autohydrolysis, Acetic Acid Pretreatment, Two-Stage Pretreatment

1. Introduction

In the palm oil mill, Oil Palm Empty Fruit Bunch (OPEFB) which constitutes 90% of biomass are discarded [1,2]. OPEFB is usually burned to generate heat and power or is converted to lower the additional value product such as compost and mulch. Even though OPEFB utilization has been conducted, this solid waste is still available in abundance. Thus, the other alternatives in the utilization of the OPEFB are required to minimize the environmental issue.

The OPEFB consists of valuable constituents such as Cellulose (19–43%) and Hemicellulose (14–37%) [3–7]. Hydrolysis of both components can generate C6 sugar (glucose) from cellulose and C 5 (xylose and arabinose) as well as C6 sugars (glucose, mannose, and galactose) from hemicellulose that these monomeric sugars are the carbon sources for fermentation to produce various fermented products like biofuel [8], biochemical [9], and biomaterials [10]. To produce such products from...
OPEFB, three general steps must be conducted, the pretreatment to degrade lignin, to reduce crystallinity, and to increase the porosity of OPEFB structure, the enzymatic saccharification (hydrolysis) to produce monomeric sugars, and the fermentation to transform such sugars to the products [11]. The more sugars are released from hydrolysis, the more product yield can be obtained. The performance of hydrolysis, moreover, is correlated to the pretreatment process [12]. Therefore, the pretreatment must be further evaluated to obtain high sugar yield.

Sugar-based production faces the problem with the complex structure of lignocellulose. Lignin, prevents cellulase to form cellulose and adsorbs the enzyme, so we must inactivate it to get more products from hydrolysis [13]. Evaluation of the pretreatment method has been numerously reported, such as physical like mechanical comminution [14], chemical like acid hydrolysis [15], physicochemical like steam explosion [16], and biological pretreatment like the use of white-rot fungus [17]. Most of the pretreatment type, however, was focusing on the way to delignify the structure of lignocellulose [18,19]. This was aimed to facilitate enzyme in degrading cellulose since the hinder component, lignin, has been removed. The phenolic compounds derived from lignin degradation would be dissolved with the other valuable sugars generated from hemicellulose after pretreatment. Because phenolic compounds were able to inhibit the further process, the liquor from these pretreatments was unable to be used [20] and detoxification was necessary [21]. The sugar also would be decomposed to be inhibitors, such as furfural and Hydroxymethyl furfural (HMF), due to severe condition and high concentration of acid pretreatment [22]. The impact of sugar decomposition was that the reducing sugar recovery will decrease [23]. The steam explosion was reported as mainly used by pretreatment methods. Once the lignocellulosic materials met an explosive decompression, they were treated with high-pressure saturated steam and suddenly being reduced. It used H2SO4 as a solvent in the condition of 170–210 °C and 5–30 minutes. The results might not be good enough in relation to composition of furfural (0.05–0.32 g component/100 g raw material) and 5-HMF (0.02–0.14 g component/100 g raw material)[24].

The purpose of this work was to obtain the best pretreatment and its corresponding condition that could recover all sugars released from cellulose and hemicellulose. The type of these monomeric sugars was reducing sugars. Autohydrolysis, acetic acid, liquor hydrolysis, and NaOH pretreatment would be identified in this work to improve reducing sugar production. The use of acetic acid was overlooked to hydrolyze cellulose and hemicellulose to its monomers [25]. Oligomeric compounds were found in spent liquor, hydrolysis through acetic acid dilution was needed [26]. NaOH pretreatment was efficient to remove lignin and it was a low inhibitor formation [27]. The use of NaOH as first stage pretreatment would be a good strategy to obtain more reducing sugar production. The second-stage pretreatment could use autohydrolysis method because it has a simple step and low cost [28].

Pretreatment at mild condition has been evaluated by several studies conducted on pretreatment of lignocellulose, including OPEFB. Harahap [29] conducted the studies with the condition of 15–60 minutes, but the profile of sugar yield was still increasing, so it must be tested on a longer time. This work would use 90 min as pretreatment time in comparison to 60 min. We looked at the condition to improve reducing sugar production from depolymerization of cellulose and hemicellulose. This pretreatment method focused on obtaining the moderate condition strategy of reducing sugar production from OPEFB. Weak acid, alkaline, and autohydrolysis were used with a variation of temperature and time strategies.

2. Material and methods

2.1. Material

The raw material, OPEFB, was obtained from PT. Condong Garut, West Java, Indonesia. OPEFB was prepared by washing with tap water followed by sun drying. Dried OPEFB was grounded using disk mill and blender to reduce the size. OPEFB is sieved to obtain particles with the maximal size of 80
mesh. Lignocellulosic composition of the OPEFB particles was identified by Chesson method [30] with the results of 48.55% cellulose, 28.06% hemicellulose, and 23.39% lignin.

2.2. Pretreatment methods

The OPEFB was pretreated with three strategies and was compared with autohydrolysis: acetic acid pretreatment, acetic acid post-treatment, and NaOH two-stage pretreatment. 10 g of solid loadings were set on 10% (g/ml) in an Erlenmeyer 300 ml. Autoclave temperature was approximately set at 115 and 125 °C and being kept at 60 and 90 min, it was counted when it reached the desired temperature. The drying temperature was set at 53–55 °C in incubator for 24 hours. Pretreated OPEFB liquor was stored in two pieces of 50 ml centrifuge tube (Biologix) and 1 ml was used for DNS method to analyze sugar reduction. This experiment was conducted in duplicate. Performance of the pretreatment method was evaluated by the yield of xylose from OPEFB and hemicellulose conversion which were calculated from the obtained concentration of xylose in the hydrolysate solution, following equation [31]:

\[
\text{Sugar yield (g/g OPEFB) = } \frac{\text{Sugar concentration} \times \text{working volume}}{\text{Initial OPEFB}}
\]  

Autohydrolysis pretreatment used water as a solvent. In acetic acid pretreatment, OPEFB was solubilized in acetic acid (Merck, p.a.) at 5 and 15% (w/w) solution. Erlenmeyer cap was made from cotton and aluminum foil, then it was heated in autoclave. Pretreated solid was dried for enzymatic hydrolysis, while liquor was analyzed with DNS method. In acetic acid post treatment, OPEFB would go through two steps. The first step was autohydrolysis. Pretreated solid was dried for enzymatic hydrolysis, while liquor was separately (1 ml) analyzed with DNS method and the other would go through the post-treatment. 30 ml of liquor was placed in Erlenmeyer and was added with 10% acetic acid to get 0.1, 0.5, and 1% (w/w) solution. DNS method was done in the end. The third strategy we used was NaOH two stage pretreatment. The first stage was a delignification of OPEFB with NaOH aqueous solution at 0.01, 0.1, and 1M. Pretreated solid was dried for second stage, while liquor was dumped. Pretreated solid then went through the autohydrolysis pretreatment. Solid fraction was dried for hydrolysis while liquor was analyzed with DNS method [29].

2.3. Hydrolysis

Enzymatic hydrolysis was conducted using Cellic® CTec2. The hydrolysis was performed in 100 mL Erlenmeyer flasks in a rotary shaker incubator that were set at 150 rpm for 72 h in room temperature. The solution was adjusted to 10% (w/v) solid to liquid ratio and pH 5 prior to hydrolysis, and 1 mL of enzyme was used in every 30 mL hydrolysis solution. Unless it was specifically indicated, the hydrolysis was conducted to the whole pretreated solution containing the pretreated OPEFB solid and the spent liquor [3].

2.4. Analytical analysis

The reducing sugar in the solution was analyzed using the UV Spectrophotometer. Aqueous dilute samples were solubilized in a solution of 25% DNS-Acid. Sample tubes were tightly capped and kept in water bath at 100 °C for 5 min. After cooling the samples, it was measured at 550 nm to determine the absorbance based on standard curve equation.

3. Results and discussion

3.1. Comparison Pretreatment Types: Autohydrolysis and Acetic Acid Pretreatment

Reducing sugar extraction by autohydrolysis was principally catalysed by acetic acid obtained from degradation of hemicellulose structure. Thus, further analysis to investigate the effect of acetic acid with the main purpose to increase reducing sugar yield was required. The result showed that the addition of acetic acid declined the amount of reducing sugar in spent liquor (figure 1). This might be
caused by the further decomposition of reducing sugar during pretreatment. The impact was the
decrease of reducing sugar recovery and inhibitor formation such as furfural and HMF. When the
temperature and time were elevated by 10 °C and 30 minutes, respectively, the reducing sugar yield in
spent liquor after autohydrolysis tended to go down. In the contrary, this from acetic acid pretreatment
increased by 0.02 g/g-OPEFB.

![Graph showing the effects of pretreatment types and the corresponding catalyst concentration on
the yield of reducing sugar on autohydrolysis and weak acid, a = pretreatment, b = hydrolysis.]

The using of this strategy was higher than Harahap [29] who obtained maximum xylose yield on 0.04
g xylose/g OPEFB. This proved that more severe condition of acetic acid could increase reducing
sugar yield. The graph shows optimum condition while temperature set on 125°C and time on 60 min.
This result may obtain because of furfural production when the pretreatment set at 125°C and 90 min,
so yield are decreased [32]. The increasing temperature of 5% acetic pretreatment from 115 °C to 125
°C led to the decline of reducing sugar yield. Conversely, significant increase of the reducing sugar
yield occurred in the higher concentration of acetic acid (15%) at 125 °C. The combination of
concentration and temperature increase positively affected to the yield. When the time was extended
from 60 to 90 minutes, however, the yield decreased for all pretreatment. In conclusion, 15% acetic
acid pretreatment gave the highest reducing sugar yield (0.40 g/g-OPEFB) as compared to two other
pretreatment.

3.2. Two stage pretreatment: autohydrolysis followed by acetic acid pretreatment
The yield of reducing sugar from autohydrolysis still could be increased by dilute acetic acid
pretreatment accompanied with heating to the spent liquor. Figure 2 showed that the yield increased
after autohydrolysed liquor was further treated by acetic acid pretreatment. The higher acetic acid
concentration was used, the more reducing sugar yield was obtained at 115 °C for 60 minutes.
Figure 2. The effects of pretreatment types and the corresponding catalyst concentration on the yield of reducing sugar on autohydrolysis and followed by weak acid, a = pretreatment, b = hydrolysis.

This result showed that the spent liquor from autohydrolysis still contained oligomer of reducing sugar. Harahap [29] used xylanase for post-treatment of autohydrolysis, and the result showed the same tendency where the reducing sugar yield increased particularly for xylose. Hence, post-treatment of the spent liquor was needed. When autohydrolysis temperature was increased by 10 °C, the maximum reducing sugar from the post-treatment was achieved at 0.5% acetic acid (0.05 g/g-OPEFB). After autohydrolysis was performed at the longer duration (up to 90 minutes), however, no significant increase occurred. From this strategy, the use of acetic acid hydrolysis of spent oligomeric compounds in liquor can increase sugar yield, but it is not significant. It may cause by intrinsic property difference of cellulose and hemicellulose [32]. Vallejos [33] got bit higher productivity of xylose from sugarcane bagasse post-treatment strategy with 0.46 g sugar/g OPEFB. He uses 3% (w/w) of H$_2$SO$_4$. Lower productivity can caused of OPEFB has lower cellulose and hemicellulose compounds and lignin is higher [34].

3.3. Two stage pretreatment: NaOH pretreatment followed by autohydrolysis

Delignification is aimed to reduce lignin contents in OPEFB. Uses of NaOH must be sufficient, or the condensation reaction will be occurs [35]. When the lignin is reduced, access to cellulose are easier and sugar yield will increase. But, uses of lignin may cause hemicellulose removal [36].
Figure 3. The effects of pretreatment types and the corresponding catalyst concentration on the yield of reducing sugar on autohydrolysis and weak alkali pretreatment, a = pretreatment, b = hydrolysis.

The results of two-stage pretreatment are shown in figure 3. First stage pretreatment effect on yield not significantly increase, but the bigger impact is obtained from hydrolysis. It may cause by ease access for enzyme to cellulose [37]. The highest reducing sugar yield from this strategy obtained 0.73 g sugar/g OPEFB from 1M NaOH + Autohydrolysis at 125°C and 60 min. Addition of NaOH concentration was significantly increase (p = 0.0031) on sugar yield, but on time and temperature are not significant (p = 0.244 and 0.375). Optimum lignin release and hemicellulose holding when OPEFB a first stage pretreated [38]. Our results higher than Zulkiple [39] has obtained fermentable sugar at 0.0439 g sugar/g OPEFB with using NaOH treatment in 10% solid loadings, 120°C and 120 min.
Table 1. Comparison on best pretreatment conditions.

| Raw Material          | Pretreatment Type | Condition | Solid Loading (g/mL) | Xylose (G. Sugar/g. material) | Glucose (g. sugar/g. material) | Reference |
|-----------------------|-------------------|-----------|----------------------|-------------------------------|-------------------------------|-----------|
| Corn Stover           | 30% ethanol with 5% H₂O₂ | 150       | 60                   | -                             | 0.238                         | [40]      |
| Populus               | 1% H₂SO₄          | 160       | 60                   | 5%                            | 0.001                         | 0.5       | [41]      |
| Wheat Straw           | Steam Explosion   | 180       | 20                   | 12%                           | 0.3001                        | 0.2576    | [42]      |
| Arundo Sugarcane Leaves | NaOH 1M          | 127       | 30                   | 10%                           | 0.1596                        | 0.0519    | [43]      |
| Sweet Sorghum         | 3% Maleic Acid    | 121       | 20                   | 6.7%                          | 0.01                           | 0.003     | [44]      |
| Rice Straw            | Popping Pretreatment | 220   | -                    | 15%                           | 0.193                         | 0.394     | [46]      |
| Cattail               | 5% NaOH           | 100       | 120                  | 10%                           | 0.41325                       | 0.07548   | [47]      |
| Norway Spruce         | 0.05% H₂SO₄       | 200       | 5                    | 3.5%                          | 0.292575                      | 0.0029975 | [48]      |
| Corn cob              | 0.25% H₂SO₄       | 120       | 30                   | 10%                           | 0.0139302                     | 0.0033339 | [49]      |
| OPEFB                 | 1M NaOH           | 125       | 60                   | 10%                           | Up to 0.73 reducing sugar     | This study|

4. Conclusions

Compared to OPEFB pretreatment using either one stage pretreatment and acetic acid two stage pretreatment, NaOH two stage pretreatment was more appropriate to be applied in particular for producing reducing sugar via enzymatic hydrolysis. The best pretreatment condition were obtained from NaOH two stage pretreatment at 125 °C for 60 min, giving the maximum reducing sugar yield of 0.73 g/g OPEFB. The difference temperature in this study showed an increased reducing sugar yield towards increased of temperature. However, difference time residence was declined the highest reducing sugar yield of the previous investigation.

References

[1] Awalludin M F, Sulaiman O, Hashim R and Nadhari W N A W 2015 An overview of the oil palm industry in Malaysia and its waste utilization through thermochemical conversion, specifically via liquefaction Renew. Sustain. Energy Rev. 50 1469–84
[2] Badan Pusat Statistik 2017 Statistik Kelapa Sawit Indonesia ed S S T Perkebunan (Jakarta: Badan Pusat Statistika)
[3] Mardawati E, Werner A, Bley T, MTAP K and Setiadi T 2014 The Enzymatic Hydrolysis of Oil Palm Empty Fruit Bunches to Xylose J. Japan Inst. Energy 93 973–8
[4] Simanungkalit S P, Mansur D, Nurhakim B, Agustin A, Rinaldi N, Muryanto and Fitriyadi M A 2017 Hydrothermal pretreatment of palm oil empty bunch AIP Conf. Proc. 1803(1) 020011–6
[5] Sudiyani Y, Styarini D, Triwahyuni E, Sudiyarmano, Sembiring K C, Aristiawan Y, Abimanyu H and Han M H 2013 Utilization of Biomass Waste Empty Fruit Bunch Fiber of Palm Oil for Bioethanol Production Using Pilot-Scale Unit Energy Procedia 32 31–8
[6] Nomanbhay S M, Hussain R and Palanisamy K 2013 Microwave-Assisted Alkaline Pretreatment and Microwave Assisted Enzymatic Saccharification of Oil Palm Empty Fruit Bunch Fiber for Enhanced Fermentable Sugar Yield J. Sustain. Bioenergy Syst. 03 7–17
[7] Palamae S, Dechatiwongse P, Choorit W, Chisti Y and Prasertsan P 2017 Cellulose and hemicellulose recovery from oil palm empty fruit bunch (EFB) fibers and production of sugars from the fibers Carbohydr. Polym. 155 491–7
[8] Kurnia J C, Jangam S V., Akhtar S, Sasmito A P and Mujumdar A S 2016 Advances in Biofuel Production from Oil Palm and Palm Oil Processing Wastes: A Review Biofuel Res. J. 3 332–46

[9] Muryanto M, Sudiyani Y and Abimanyu H 2016 Optimasi Proses Perlakuan Awal NaOH Tandan Kosong Kelapa Sawit untuk menjadi Bioetanol J. Kim. Terap. Indones. 18 27–35

[10] Kresnowati M, Marduwati E and Setiadi T 2015 Production of Xylitol from Oil Palm Empty Friuts Bunch: A Case Study on Bioefinery Concept Mod. Appl. Sci. 9(7) 206–13

[11] Nurul Adela B, Nasrin A B, Loh S K and Choo Y M 2014 Bioethanol Production by Fermentation of Oil Palm Empty Fruit Bunches Pretreated with Combined Chemicals J. Appl. Environ. Biol. Sci. 4 234–42

[12] Łukajtis R, Kucharska K, Holowacz I, Rybarczyk P, Wychodnik K, Słupek E, Nowak P and Kamiński M 2018 Comparison and optimization of saccharification conditions of alkaline pre-Treated triticale straw for acid and enzymatic hydrolysis followed by ethanol fermentation Energies 11(3) 639

[13] Robak K and Balcerek M 2018 Review of Second Generation Bioethanol Production from Residual Biomass Food Technol. Biotechnol. 56 174–87

[14] Kumar P, Barrett D M, Delwiche M J and Stroeve P 2009 Methods for pretreatment of lignocellulosic biomass for efficient hydrolysis and biofuel production Ind. Eng. Chem. Res. 48 3713–29

[15] Wen P, Zhang T, Wang J, Lian Z and Zhang J 2019 Production of xylooligosaccharides and monosaccharides from poplar by a two-step acetic acid and peroxide/acetic acid pretreatment Biotechnol. Biofuels 12 1–13

[16] Seidel C M, Brethauer S, Gyenge L, Rudolf Von Rohr P and Studer M H 2019 Two-stage steam explosion pretreatment of softwood with 2-naphthol as carbocation scavenger Biotechnol. Biofuels 12 37

[17] Ding C, Wang X and Li M 2019 Evaluation of six white-rot fungal pretreatments on corn stover for the production of cellulolytic and ligninolytic enzymes, reducing sugars, and ethanol Appl. Microbiol. Biotechnol 103(14) 5641–52

[18] Yu Q, Liu R, Li K and Ma R 2019 A review of crop straw pretreatment methods for biogas production by anaerobic digestion in China Renew. Sustain. Energy Rev. 107(C) 51–8

[19] Wang J, Chen X, Chio C, Yang C, Su E, Jin Y, Cao F and Qin W 2019 Delignification overmatches hemicellulose removal for improving hydrolysis of wheat straw using the enzyme cocktail from Aspergillus niger Bioreour. Technol. 274 459–67

[20] Pienkos P T and Zhang M 2009 Role of pretreatment and conditioning processes on toxicity of lignocellulosic biomass hydrolysates Cellulose 16 743–62

[21] Lee K M, Min K, Choi O, Kim K-Y, Woo H M, Kim Y, Han S O and Um Y 2015 Electrochemical detoxification of phenolic compounds in lignocellulosic hydrolysate for Clostridium fermentation Bioprocess. Technol. 187 228–34

[22] Jönsson L J, Alriksson B and Nilvebrant N-O 2013 Bioconversion of lignocellulose: inhibitors and detoxification Leif Biotechnol. Biofuels 6(1) 16

[23] Jiang T-T, Liang Y, Zhou X, Shi Z-W and Xin Z-J 2019 Optimization of a pretreatment and hydrolysis process for the efficient recovery of recycled sugars and unknown compounds from agricultural sweet sorghum bagasse stem pith solid waste PeerJ 6 e 6186

[24] Alviria P, Negro M J, Ballesteros I, González A and Ballesteros M 2016 Steam Explosion for Wheat Straw Pretreatment for Sugars Production Bioethanol 2 66–75

[25] Keskin T, Nalakath Abubackar H, Arslan K and Azbar N 2019 Chapter 12 - Biogas Production From Solid Wastes Biomass, Biofuels, Biochemicals ed Lee D J, Pandey A et al (Amsterdam: Elsevier)

[26] Agudelo R A, García-Aparicio M P and Görgens J F 2016 Steam explosion pretreatment of triticale (× Triticosecale Wittmack) straw for sugar production N. Biotechnol. 33 153–63
[27] Brodeur G, Yau E, Badal K, Collier J, Ramachandran K B and Ramakrishnan S 2011 Chemical and Physicochemical Pretreatment of Lignocellulosic Biomass: a Review. Enzyme Res 2011 787532

[28] Han Q, Jin Y, Jameel H, Chang H min, Phillips R and Park S 2014 Autohydrolysis Pretreatment of Waste Wheat Straw for Cellulosic Ethanol Production in a Co-located Straw Pulp Mill Appl. Biochem. Biotechnol. 175 1193–210

[29] Harahap B M and Kresnowati M T A P 2018 Moderate pretreatment of oil palm empty fruit bunches for optimal production of xylitol via enzymatic hydrolysis and fermentation Biomass Convers. Biorefinery 8 255–63

[30] Datta R 1981 Acidogenic Fermentation of Lignocellulose–Acid Yield and Conversion of Components Biotechnol. Bioeng. 23 2167–70

[31] Selig M, Weiss N and Ji Y 2008 Enzymatic Saccharification of Lignocellulosic Biomass: Laboratory Analytical Procedure (LAP) NREL (Golder, Colo.: National Renewable Energy Laboratory)

[32] Rabemanolontsoa H and Saka S 2016 Various pretreatments of lignocellulosics Bioresour. Technol. 199 83–91

[33] Vallejos M E, Chade M, Mereles E B, Bengoechea D I, Brizuela J G, Felissia F E and Area M C 2016 Strategies of detoxification and fermentation for biotechnological production of xylitol from sugarcane bagasse Ind. Crop. Prod. 91 161–9

[34] Balasundram V, Alias N, Ibrahim N, Md. Kasmani R, Isha R, Abd. Hamid M K and Hasbullah H 2018 Thermal Characterization of Malaysian Biomass via Thermogravimetric Analysis J. Energy Saf. Technol. 1 31–8

[35] Chen H, Wang L, Chen H and Wang L 2017 Pretreatment Strategies for Biochemical Conversion of Biomass Technologies for Biochemical Conversion of Biomass 1st ed (Cambridge, Massachusetts: Academic Press) 21–64

[36] Geng W, Richard A V, Pawlak J J and Chang H 2018 Effect of Delignification on Hemicellulose Extraction from Switchgrass, Poplar, and Pine and Its Effect on Enzymatic Convertibility of Cellulose-rich Residues BioRes. 13(3) 4946–63

[37] Paixao S M, Ladeira S A, Silva T P, Arez B F, Roseiro J C, Martins M L L and Alves L 2016 Sugarcane bagasse delignification with potassium hydroxide for enhanced enzymatic hydrolysis RSC Adv. 6 1042–52

[38] Mardawati E, Badruzaman I, Nurjanah S and Bindar Y 2018 Effect of organosolv pretreatment on delignification for bioethanol feedstock from oil palm empty fruit bunch (OPEFB) IOP Conf. Ser.: Earth Env. Sci. 209 012009

[39] Zulkiple N, Maskat M Y and Hassan O 2016 Pretreatment of Oil Palm Empty Fruit Fiber (OPEFB) with Aquaeous Ammonia for High Production of Sugar Procedia Chem. 18 155–61

[40] Park Y C, Kim J S and Kim T H 2018 Pretreatment of corn stover using organosolv with hydrogen peroxide for effective enzymatic saccharification Energies 11 1–9

[41] Meng X, Wells T, Sun Q, Huang F and Ragauskas A 2015 Insights into the effect of dilute acid, hot water or alkaline pretreatment on the cellulose accessible surface area and the overall porosity of Populus Green Chem. 17 4239–46

[42] Eisenhuber K, Krennhuber K, Steinmüller V and Jäger A 2013 Comparison of different pretreatment methods for separating hemicellulose from straw during lignocellulose bioethanol production Energy Procedia 40 172–81

[43] Lemões J S, Lemons e Silva C F, Avila S P F, Montero C R S, Silva S D dos A e., Samios D and Peralba M do C R 2018 Chemical pretreatment of Arundo donax L. for second-generation ethanol production Electron. J. Biotechnol. 31 67–74

[44] Suhardi V S H, Prasai B, Samada H and Boopathy R 2013 Evaluation of pretreatment methods for lignocellulosic ethanol production from energy cane variety L 79–1002 Int. Biodeterior. Biodegrad. 85 683–7
[45] Oktaviani M, Hermiati E, Thontowi A, Laksana R P B, Kholida L N, Andriani A, Yopi and Mangunwardoyo W 2019 Production of xylose, glucose, and other products from tropical lignocellulose biomass by using maleic acid pretreatment *IOP Conf. Ser. Earth Environ. Sci.* **251** 012013

[46] Wi S G, Choi I S, Kim K H, Kim H M and Bae H J 2013 Bioethanol production from rice straw by popping pretreatment *Biotechnol. Biofuels* **6**(1) 166

[47] Sopajarn A and Sangwichien C 2015 Optimization of Enzymatic Saccharification of Alkali Pretreated Typha angustifolia for Glucose Production *Int. J. Chem. Eng. Appl.* **6** 232–6

[48] Kuittinen S, Rodriguez Y P, Yang M, Keinänen M, Pastinen O, Siika-aho M, Turunen O and Pappinen A 2016 Effect of Microwave-Assisted Pretreatment Conditions on Hemicellulose Conversion and Enzymatic Hydrolysis of Norway Spruce *Bioenergy Res.* **9** 344–54

[49] Zhu Y, Huang J, Sun S, Wu A and Li H 2019 Effect of Dilute Acid and Alkali Pretreatments on the Catalytic Performance of Bamboo-Derived Carbonaceous Magnetic Solid Acid *Catalysts* **9** 245