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Hydrothermal and wet disk milling pretreatment for high conversion of biosugars from oil palm mesocarp fiber

Mohd Rafein Zakaria [a,b,*], Mohd Nor Faiz Norrahim [b], Satoshi Hirata [a], Mohd Ali Hassan [b,c]

[a] Biomass Refinery Research Center, National Institute of Advanced Industrial Science and Technology (AIST), 3-11-32 Kagamiyama, Higashi-Hiroshima, Hiroshima 739-0046, Japan
[b] Department of Bioprocess Technology, Faculty of Biotechnology and Biomolecular Sciences, Universiti Putra Malaysia, 43400 UPM Serdang, Selangor, Malaysia
[c] Department of Process and Food Engineering, Faculty of Engineering, Universiti Putra Malaysia, 43400 UPM Serdang, Selangor, Malaysia

HIGHLIGHTS

- Combined pretreatment using hydrothermal and WDM was performed on OPMF.
- Maximal H_2O contact and hemicellulose removal were favored under HCW than SHS.
- Defibrillation of OPMF is key factor for higher glucose conversion.
- Optimal combined pretreatment of HCW–WDM was 180 °C, 20 min after 9 cycles of WDM.
- Glucose yield (98%) obtained from HCW–WDM with power requirement 9.6 MJ/kg OPMF.

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ABSTRACT

Eco-friendly pretreatment methods for lignocellulosic biomass are being developed as alternatives to chemical based methods. Superheated steam (SHS), hot compressed water (HCW) and wet disk milling (WDM) were used individually and with combination to partially remove hemicellulose and alter the lignin composition of recalcitrant structure of oil palm mesocarp fiber (OPMF). The efficiency of the pretreatment methods was evaluated based on the chemical compositions altered, SEM analysis, power consumption and degree of enzymatic digestibility. Hemicellulose removal (94.8%) was more pronounced under HCW compared to SHS, due to maximal contact of water and production of acetic acid which enhanced further degradation of hemicellulose. Subsequent treatment with WDM resulted in defibrillation of OPMF and expansion of the specific surface area thus increasing the conversion of cellulose to glucose. The highest glucose yield was 98.1% (g/g-substrate) when pretreated with HCW (200 °C, 20 min) and WDM which only consumed 9.6 MJ/kg of OPMF.

1. Introduction

Malaysia annually generates 6.93 million tonnes of oil palm biomass (dry basis) (Nazir et al., 2013) which provides potential bioresources for the conversion into value-added products such as chemical feedstocks, biosugars, biofuels, bioplastic, cellulose and composite production (Zahari et al., 2014). The other major resource being wasted is excess steam produced through burning of oil palm oil biomass (Yoshizaki et al., 2013). The integration and effective utilization of biomass and excess steam are believed to provide eco-processes and green products towards a sustainable oil palm industry. Oil palm biomass has attracted attention of researchers due to its potential utilization for sugar recovery. Oil palm empty fruit brunches (OPEFB), oil palm mesocarp fiber (OPMF) and oil palm frond fiber (OPFF) can be obtained abundantly throughout the year. Extensive research has been done on conversion of these materials to ethanol by hydrolysis of cellulose to fermentable sugars (Bakar et al., 2012; Zahari et al., 2014). The presence of hemicellulose and lignin hinders the access of the cellulase to cellulose, thus resulting in low efficiency of the hydrolysis (Sun and Cheng, 2012). Previous reports have shown that, by removing hemicellulose and lignin (Zakaria et al., 2014b), reduction of cellulose crystallinity (Hideno et al., 2009; Silva et al., 2010) and increase of porosity (Lee et al., 2010) from pretreatment processes can substantially improve the enzymatic hydrolysis.

* Corresponding author at: Biomass Refinery Research Center, National Institute of Advanced Industrial Science and Technology (AIST), 3-11-32 Kagamiyama, Higashi-Hiroshima, Hiroshima 739-0046, Japan. Tel./fax: +81 82 420 8309.
E-mail address: rafein-zakaria@aist.go.jp (M.R. Zakaria).

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Selection of suitable pretreatment is necessary to get rid of this recalcitrance prior to saccharification for bioethanol and biofuels production (Mosier and Wyman, 2005; Weiqi et al., 2013). Efficient utilization of lignocellulose requires an initial pretreatment step to minimize the barrier caused by hemicellulose and lignin, thus higher enzymatic digestibility can be achieved (Vancov and McIntosh, 2012). Several pretreatments can be applied to oil palm biomass such as chemical, mechanical and hydrothermal treatment in order to overcome this hindrance. Pretreatment involving hot compressed water (HCW), liquid hot water (LHW), steam, and superheated steam (SHS) are gaining interest recently, as these treatments are more environmental friendly, and time saving for enzymatic saccharification of natural biomass (Yu et al., 2011; Inoue et al., 2008; Weiqi et al., 2013; Yunos et al., 2012; Nik Mahmud et al., 2013). Superheated steam is unsaturated (dry) steam generated by the addition of sensible heat to saturated (wet) steam (Bahrin et al., 2012). Pretreatment using SHS has several advantages such as improved energy efficiency, higher drying rates, use of an air-free environment, and reduced environmental impact when condensate is reused (Nik Mahmud et al., 2013). Moreover, SHS pretreatment is a cost-effective method for large-scale purpose and it is one of the eco-friendly approaches due to the utilization of excess steam produced at the palm oil mill. However, SHS or other hydrothermal treatment alone does not produce a high yield of hydrolyzed sugars (Nik Mahmud et al., 2013; Bahrin et al., 2012; Zakaria et al., 2014b). Combination of hydrothermal pretreatment with other mechanical pretreatment has been reported to substantially improve sugar yields (Inoue et al., 2008; Hideno et al., 2012).

Comminution processes such as planetary/attrition ball milling and wet disk milling have resulted in reduction of particle size, increased surface area, increased pore volume and reduced crystallinity (CrI) of cellulose, thus enhancing the enzymatic digestibility of biomass (Hideno et al., 2009; Silva et al., 2010; Zakaria et al., 2014a). There have been very few reports of pretreatment technologies tested on OPMF. The effect of combined pretreatment using alkaline hydrothermal and ball milling to enhance enzymatic hydrolysis of OPMF was reported (Zakaria et al., 2014b). Even though high recovery of xylose and glucose were obtained, higher energy consumption was recorded from ball mill process. Recently, wet disk milling (WDM) has been used to produce relatively low levels of inhibitors and increased degree of defibrillation which created more space between microfibrils, thus enhanced the enzymatic hydrolysis of the fiber (Lee et al., 2010; Zhang et al., 2013). Wet disk milling should be combined with other treatments in order to facilitate the enzyme accessibility of the fiber (Gao et al., 2012). Combination of hydrothermal (LHW and or HCW) with WDM pretreatment on rice straw (Hideno et al., 2012) and eucalyptus (Weiqi et al., 2013) have shown an improvement of sugar yield compared to hydrothermal pretreatment alone. Compared to previous studies on combination of HCW and WDM the present study highlighted a suitability of OPMF as raw material for high glucose conversion comparable to rice straw and eucalyptus chips. In addition, a bigger space gap between upper and lower grinders (150–300 μm) was adopted in the present study compared to 20–40 μm (Hideno et al., 2012; Weiqi et al., 2013) in order to avoid overheating of samples and additional maintenance cost of grinders.

Therefore, in this work we investigate the suitability of individual hydrothermal (SHS and HCW) and WDM pretreatment alone and its combination on OPMF in order to understand the mechanism involved for each method used that affects biomass structure and cellulose properties. The effect of SHS, HCW and WDM on size reduction, specific surface area, morphological analysis and energy consumption are compared and discussed.

2. Methods

2.1. Raw materials

The OPMF used in this study was obtained from Seri Ulu Langat Palm Oil Mill, Dengkil, Selangor, Malaysia. The samples were sun dried and cut by milling cutters to size <2 mm. The ground samples were stored in *vacuo* at room temperature (24 °C) prior to further analysis.

2.2. Hydrothermal pretreatment

2.2.1. Superheated steam (SHS)

The SHS pretreatment of OPMF was carried out using a lab scale superheated steam machine (Naomoto Co., Tokyo, Japan) with outer and inner oven sizes of (W70 cm × D60 cm × H60 cm) and (W20 cm × D30 cm × H20 cm), respectively. The SHS oven consists of a stainless steel heating chamber and a heater kind of boiler. Steam was generated by electric steam generator (NBC-2070R model, Naomoto Co., Tokyo, Japan) with steam evaporation and steam pressure at 10.5 kg/h and 0.3 MPa, respectively. Tap water was pretreated by auto-softener machine (Mk-6 J model, Maruyama Co., Ltd., Tokyo, Japan) and controlled by valve timer. Approximately 0.9–1.0 kg of ion exchange resin-salt was used per regeneration of soft-water. The selection of pretreatment conditions by SHS for OPMF was based on the best treatment condition reported in the literature (Nordin et al., 2013), conducted at temperature ranging from 190 °C to 230 °C for 60 min.

2.2.2. Hot compressed water (HCW)

Hot compressed water pretreatment was performed using a 1 L stainless steel autoclave (Nitto Koatsu Co., Tsukuba, Japan) as reported earlier (Hideno et al., 2009) with slight modification. Oil palm mesocarp fiber (50 g) and water (500 mL) were added to autoclave system and mixed at 250–300 rpm and the samples mixture were heated to a reaction temperature range from 160 °C to 200 °C at a heating rate of 2–3 °C/min and maintained for 20 min. The autoclave was left and cooled down at room temperature prior to further process. The pretreated solids were separated from slurries by using Advantec No. 2 membrane filter and rinsed with distilled water until neutral pH.

2.2.3. Wet disk milling (WDM)

Wet disk milling was performed using Supermass colloider MKZA10 (Masuko Saangyo Co., Ltd., Saitama, Japan) as reported earlier (Hideno et al., 2009) with slight modification. Briefly, the ceramic nonporous disk grinders was adjusted for a clearance of 150–300 μm between the upper and lower grinders and revolved at 1800 rpm. Fifty (50) gram of untreated (<2 mm) and hydrothermally pretreated OPMF (SHS and HCW) solids were soaked in water (1 L) for 2–3 days prior to WDM and the operation was repeated for 2–20 times. The pretreated slurry was centrifuged at 10,000×g for 15 min and the solids recovered were subjected to freeze-drying prior to enzymatic hydrolysis and other analysis.

2.3. Enzymatic hydrolysis

Unless otherwise stated, enzymatic hydrolysis was performed using an enzyme cocktail constituting 40 FPU/mL *Acremonium* cellulase (Meiji Seika Co, Japan), and 10% Optimash BG (Genencor International, California, USA) as reported previously (Zakaria et al., 2014b). The enzymatic hydrolysis was performed at 50 °C for 72 h with stirring/shaking. The experiment was performed in triplicates and the results are presented as the average values.