

Hydrothermal and Wet Disk Milling Pretreatment for Biosugar and Nanofiber Production From Oil Palm Mesocarp Fiber

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Introduction

Selection of suitable pretreatment is necessary to get rid of biomass recalcitrance before saccharification for bioethanol and biofuels production (Weiqi et al., 2013). Recently, pretreatment involving hot compressed water (HCW), liquid hot water (LHW), steam, and superheated steam (SHS) are gaining interest. These treatments are environmentally friendly and time-saving for enzymatic saccharification of natural biomass (Yu et al., 2011; Weiqi et al., 2013).

Recently, wet disk milling (WDM) has been used to produce relatively low levels of inhibitors and increased the 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 to facilitate the enzyme accessibility of the fiber (Gao et al., 2012). The 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.

Therefore, in this work, we investigate the suitability of individual hydrothermal and WDM pretreatment alone and its combination on OPMF to understand the mechanism involved for each method used that affects biomass structure and cellulose properties Figure 1.

Individual hydrothermal (SHS and HCW) and WDM pretreatment and their combinations have resulted in morphological changes of OPMF as evaluated by SEM. Figure 2a shows that the untreated OPMF has a rigid and intact surface which impedes the penetration of degradation enzymes to the targeted compounds. A small morphological alteration was observed after being treated by SHS at 200°C, 60 min Figure 2b. In contrast, Figure 2c showed a higher degree of surface morphology alteration when OPMF was pretreated with HCW, whereas efficient dissolution of hemicellulose may result in peeling-off of outer layer of fiber (Zakaria et al., 2014b). Wet disk milling was performed to reduce particle size and increased specific surface area. Figure 2d shows the SEM image of WDM treated-OPMF samples after 20 operation cycles. From Figure 2e-f, there is no significant difference from combined hydrothermal (SHS and HCW) and WDM in comparison to WDM pretreatment alone. Serial dilution was performed, washed several times with ethanol as described in the methodology section to observe the differences between SHS and HCW-WDM pretreatment. The degree of defibrillation and particle size reduction played a critical part to enhance the conversion of hydrolysed sugars from lignocellulosic material. From the morphological analysis, defibrillation of OPMF occurred rapidly as the amount of operation cycle of WDM increased. Well-defibrillated fibers with a diameter size smaller than 50 nm were observed for OPMF treated

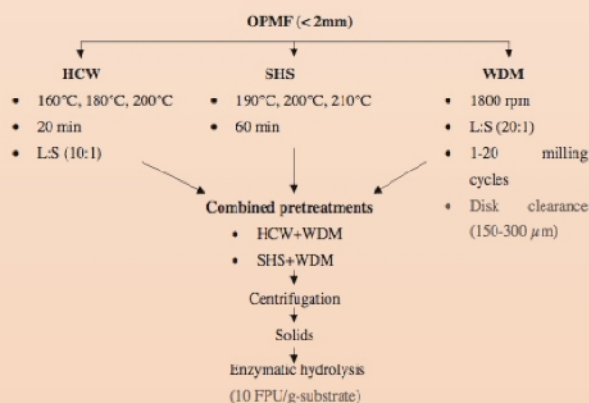


Fig. 1. Schematic diagram of pretreatments on OPMF samples for biosugars and nanofibers production.

with HCW as shown in Figure 2h-i. These results indicate the defibrillation of OPMF by HCW pretreatment is more pronounced than defibrillation by SHS pretreatment Figure 2g probably due to greater hemicellulose removal, which resulted in increased specific surface area of the particles. Partial removal of hemicellulose and alteration or migration of lignin from the cellulose-hemicellulose-lignin matrix may have caused OPMF to be more ductile and led to loosening packing of cellulose microfibrils (Lee et al., 2010). Thus, it will ease the WDM pretreatment by reducing the particle size and increased the efficiency of hydrolytic enzyme penetration to the fiber (Weiqi et al., 2013).

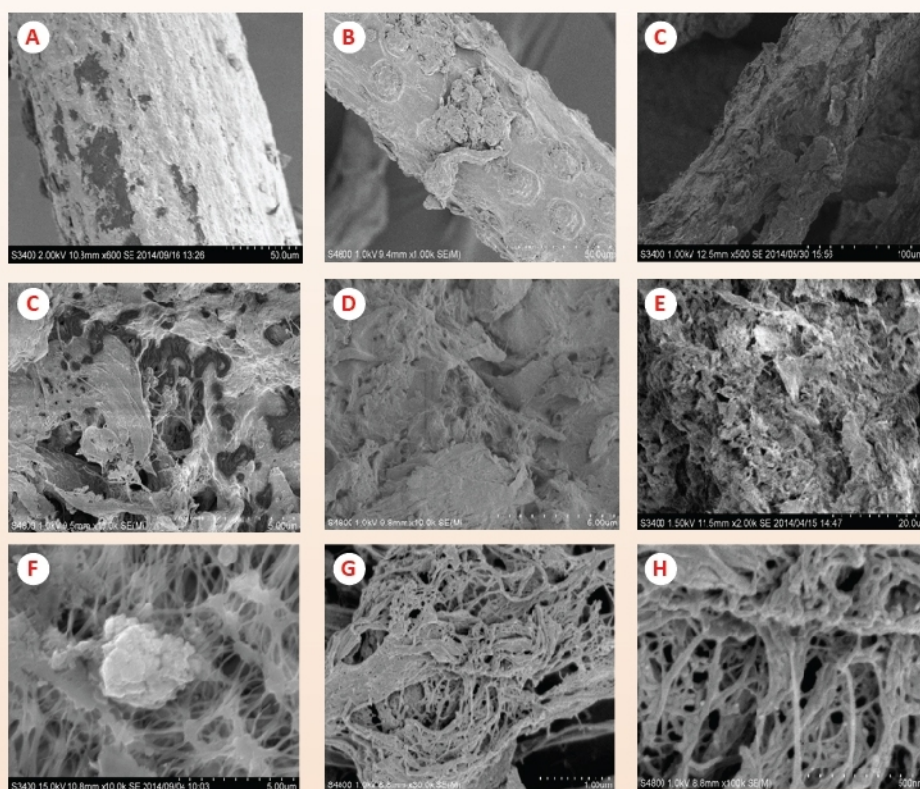


Fig. 2. a) Untreated OPMF, b) SHS (200°C, 60 min)-treated, c) HCW (200°C, 60 min)-treated, d) WDM-treated, e) SHS (200°C, 60 min)-WDM_{cycle9}-treated, f) HCW (200°C, 60 min)-WDM_{cycle9}-treated, g) diluted (0.01%) SHS (200°C, 60 min)-WDM_{cycle9}-treated, h) diluted (0.01%) HCW (200°C, 20 min)-WDM_{cycle9}-treated, i) enlarge image of (h)

Table 1 shows the comparative analysis of yield of hydrolyzed sugars and energy consumption from combined WDM with HCW pretreatments at different pretreatment severities with other studies. Wet disk milling pretreatment alone required about 18.9 MJ/kg of OPMF and recorded higher in comparison to rice straw, sugarcane bagasse and sugarcane straw (Hideno et al., 2009; Silva et al., 2010). Therefore, WDM pretreatment required lower energy consumption compared to ball milling pretreatment (Zakaria et al., 2014a; Zakaria et al., 2014b). It was observed that the energy requirement was decreased when OPMF was induced with hydrothermal pretreatment before WDM treatment, which resulted in energy consumption of 17.5 MJ/kg of

OPMF when pretreated with HCW at 200°C, 20 min). The yield of hydrolysed sugars correlated with the WDM processing cycles. Based on the current study, the energy consumption depended on the types of pretreatment, as partial removal of hemicellulose and migration of lignin are sufficient to weaken the matrix of the lignocellulosic network between cellulose microfibrils. It was also observed that the higher amount of energy consumption of WDM depended on the viscosity of the sample where HCW-treated OPMF was found to have the higher degree of viscosity than SHS-treated OPMF. By considering the higher specific surface area and degree of defibrillation from the HCW-WDM process, lower enzyme loading may apply.

Table 1. Comparative analysis of yield of hydrolyzed sugars and energy consumption from SHS and HCW at different treatment severities with other studies.

Sample	Yield of glucose,% (g/g-substrate)	of	Energy consumption (MJ/kg biomass)	References
OPMF WDM _{9cycle}	30.2 ± 2.4		18.9	This study
HCW _{180°C, 20 min} + WDM _{9cycle}	86.2 ± 1.4		14.9	This study
HCW _{200°C, 20 min} + WDM _{9cycle}	91.1 ± 4.3		17.5	This study
WDM _{10cycle} -rice straw	78.5 ± 5.6		5.4	Hideno et al. (2009)
WDM _{10cycle} -sugarcane bagasse	31.5 ± 1.7		10.6	Silva et al. (2010)
WDM _{10cycle} -sugarcane straw	56.1 ± 0.5		14.8	Silva et al. (2010)
LHWP _{180°C, 20 min} + WDM _{15cycle} -eucalyptus	91.6			Weiqi et al. (2013)
HCW _{135°C, 60 min} + WDM _{15cycle} -rice straw	90.0			Hideno et al. (2012)

* WDM= wet disk milling, SHS= superheated steam, HCW= hot compressed water, and LHWP= liquid hot water pretreatment.

Conclusions

Hydrothermal pretreatment alone showed an increase in degree of enzymatic digestibility compared to untreated OPMF. Hemicellulose removal was more pronounced in HCW, and subsequent WDM treatment has promoted defibrillation of cellulose microfibril, hence increased specific surface area. Furthermore, combined HCW and WDM offered shorter milling cycles and lower power consumption with more than 98% of glucose yield. To the best of our knowledge, this is the first report of eco-friendly combined pretreatment using hydrothermal and WDM to increase the enzymatic efficiency of OPMF and cost efficient for nanofiber production.

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