DELIGNIFICATION OF PALM FIBER BY MICROWAVE ASSISTED CHEMICAL PRETREATMENT FOR IMPROVING ENERGY EFFICIENCY

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ABSTRACT Fossils fuels are main source of energy. However, their depletion has caused widespread concern. In recent years, utilization of agricultural wastes for energy has received a lot of attention. Lignocellulosic biomass is an abundantly available agricultural waste. It is a candidate for economic and environmental friendly source of biofuel production. However, high lignin content of the biomass reduces its digestibility and energy recovery. Suitable pretreatment of biomass can degrade the lignin content and enhance its conversion to biofuel. This work studied the effects of various microwave assisted chemical pretreatments on delignification of palm fiber to improve energy recovery. A total of 12 solutions using four chemicals, namely, sulphuric acid, sodium hydroxide, hydrogen peroxide, and sodium carbonate with 3 different concentrations i.e. 2%, 3.5%, and 5% were prepared. 1 g of palm fiber was pretreated in 10 ml of each solution, for 48 h. The solution along with the biomass was then heated by microwave irradiation for 5 min. The results showed that the pretreatments performed were effective in reducing lignin content and increasing cellulose content of the samples. Sodium hydroxide and hydrogen peroxide were most suitable for delignification of palm fiber. Treatment with these chemicals reduced the lignin content and changed the C/N ratio to the optimum range. This increased cellulose content enhances energy potential to produce biofuel by conversion to ethanol or biogas.

(**Keywords:** Chemical pretreatment, Microwave pretreatment, Palm fibre, Lignocellulosic Biomass, Biomass Waste, Waste to energy)

INTRODUCTION

The world is using fossil fuels as the main source of energy, however their reserves are depleting rapidly due to increased energy demand, and also the usage and derivation of fossil fuels produce pollutants that are harmful to the environment. These considerations demand an energy source, that is economical, abundantly available, sustainable, and environment friendly (Demirbas et al., 2011; Escamilla-Alvarado et al., 2012).

Agricultural wastes (biomass) are viewed as good candidates for alternative energy sources. With an estimated worldwide annual production of 140 billion metric tonnes, agricultural wastes have the potential to produce energy approximately equivalent to 50 billion tonnes of oil. Energy produced from biomass is environment friendly and carbon neutral. It also reduces our dependence on fossil fuels, thereby contributes to energy security and clean climate change mitigations (Compendium 2009).

Biofuel production from various types of solid wastes and residues, like municipal and agricultural solid wastes, by fermentation and anaerobic digestion has been experimentally investigated and is considered to be a viable future options available, due to its economic feasibility (Chandra et al., 2012; Escamilla-Alvarado et al., 2010).

This agricultural biomass waste is called lignocellulosic biomass and contains polymers like cellulose and hemicellulose, which are easily degraded and converted into simple monomers and sugars, followed by conversion to biogas, by anaerobic digestion. However, the hemicellulose and cellulose are densely packed by the layers of lignin, which poses as a protective wall in plant materials and restricts digestion. Thus for successful and rapid biodegradation or anaerobic digestion it is necessary to pretreat lignocellulosic biomass as shown in Fig.1. Biomass pretreatment or delignification, increases the surface area and porosity of biomass, and decreases the crystallinity of cellulose and hemicellulose and the degree of polymerization.

Various pretreatment techniques are classified into three main categories as;

1. Mechanical or Physical: This treatment involves milling, grinding, irradiation, thermal, hydrothermal, and other physical techniques.

2. Chemical and Physico Chemical: This treatment involves mixing the feedstock with chemicals, or soaking in chemical solutions, like acid, alkali, oxidizing agents. It also includes gas

treatment, steam explosion, and solvent extraction techniques.

3. Biological: This treatment involves introducing fungi and/or actinomycetes to the feedstock, which treat the biomass waste biologically (Chandra et al., 2012; Hendriks & Zeeman, 2009).

All the pretreatments are effective in treating lignocellulose, but the degree of treatment varies widely depending upon the method selected. Previous studies have shown that chemical or physico chemical treatment yield better results in treating lignocellulose feedstocks as compared to other treatments (Hendriks & Zeeman, 2009).

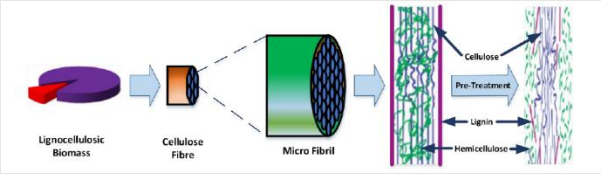

Figure 1-Pretreatment Process of Lignocellulosic Biomass (Chandra et al., 2012)

Chemical pretreatment is the most widely studied, and useful technique for lignocellulosic biomass degradation. It involves several treatment methods, but most effective are acid and alkaline pretreatments. The concentration of the acidic or the alkaline solution varies, but in most cases dilute acidic and alkaline pretreatments are performed so that it must only be sufficient to disrupt the lignin content, thereby increasing the cellulose content; higher concentration may cause loss of cellulose, which is to be degraded into biofuels. Hsu et al. (2010) reported increase in cellulose content to more than 50% using 0.5 to 1% sulphuric acid pretreatment of rice straw. Park and Kim (2012) compared various alkaline pretreatments to evaluate their effects on lignin disruption and enzymatic digestibility of various lignocellulosic biomass (Eucalyptus residue, Larix leptolepis, Pinus rigida, rice straw and barley straw); they found that alkaline pretreatments were effective in lignin degradation.

Huge amounts of biomass waste are generated by the palm oil industry in various parts of the world including, Asia, Africa, and South America. Oil palm is originally an African crop, however, in the 19th century this crop was introduced to South East Asia, which currently holds the most oil palm cultivation worldwide. Largest producers of oil palm are Malaysia and Indonesia, which jointly produce about 85% of worldwide palm oil (Sulaiman et al., 2011).

Palm trees are used to produce edible oil, which is extracted from the fruits of these trees. Palm oil has many uses ranging from food industry to industrial applications due to its moderate price and semi-liquid status. The waste generated from palm oil industry is also useful. Palm oil waste generated is roughly estimated to be around 4 kg waste for each kg of palm oil produced (Sulaiman et al., 2011). This waste, especially empty fruit bunch (EFB) has good potential to be used for energy generation due to its high polysaccharide content (Chiesa & Gnansounou, 2014). It has been used to produce biofuels mainly bioethanol.

The high cellulose content of empty fruit bunches can be utilized to produce bioethanol by fermentation, or converting it into biogas by anaerobic digestion. The polysaccharide polymers present in EFB are broken down by enzymes to fermentable sugars, but unfortunately this cellulose content is located in cell walls protected by a lignin layer, which due to its recalcitrant nature reduces the enzyme contact with cellulose and thereby reduces digestion and fermentation efficiency. In order to avoid this resistance, it becomes necessary to pretreat this lignocellulosic material before its conversion into biofuel.

EFB has been used to produce bioethanol by using different methods (Hassan et al., 2013; Ming J. Lau et al., 2010; Park et al., 2013). Studies suggest that pretreatments have played an effective role in increasing the cellulose conversion efficiency, and thereby increasing biofuel production. Hassan et al. (2013) studied the pretreatment effects of steam assisted 5% acidic and alkaline pretreatments by using acetic acid as well as NaOH solutions and found that steam assisted acetic acid produced better results for enzymatic saccharification and sugar production enhanced to 62.36% xylose and 81.84% glucose. Han et al. (2011) studied the effect of alkaline pretreatment and used concentrated NaOH (3M). They reported 85% total cellulose conversion by enzymatic saccharification. Park et al. (2013) studied the effect of milder concentration of NaOH (1M) on ethanol concentrations. They reported production of ethanol concentrations higher than 40g/L by simultaneous saccharification and fermentation of NaOH-pretreated EFBs at 121°C.

Studies suggest that microwave pretreatment is also very effective in delignification of lignocellulosic biomass; whether used individually or in combination with chemical pretreatment (Laghari et al., 2014). Chang et al. (2011) found microwave irradiation very effective over conventional thermal treatment for waste sludge. Zhu et al. (2005) used microwave

assisted alkali pretreatment for rice straw and reported increase in cellulose content from 38.9% to 69.3% with lignin reduction from 13.6 to 5%. Chen et al. (2011) studied sugarcane bagasse lignocellulose disruption by using microwave assisted sulphuric acid, and reported increase in cellulose content from 52.25% to 67.31%.

The present study was conducted to evaluate the effects of microwave assisted acidic and alkaline pretreatments on EFB palm fiber, in terms of delignification, and to determine suitable pretreatment conditions to obtain high cellulose yield.

MATERIALS & METHODS

Materials

Palm empty fruit bunch was obtained from local farmers at Tronoh, fiber from EFB was milled and ground to a size of 1 mm or less using a Rocklabs mortar grinder type BTRM Model 1A. After milling the sample was placed in air tight containers for later use.

Chemical Solutions

A total of 12 solutions using four chemicals, sulphuric acid, sodium hydroxide, hydrogen peroxide, and sodium carbonate with 3 different concentrations 2%, 3.5%, and 5% were prepared. Strong solutions of sodium hydroxide and sodium carbonate were prepared using solid pallets, and then they were diluted. 1 g of extractive free EFB palm fiber was pretreated in 10 ml of each solution, for 48 h. The solution along with the biomass was then heated by microwave irradiation for 5 min.

Microwave

Faber Microwave Oven Model FMO 7020 with input power 1050 W and output power 700 watt was used. This Microwave oven had 5 settings as Low, Medium Low, Medium, Medium High, and High, with output of 17%, 33%, 55%, 77%, and 100% microwave energy respectively. In this study Medium Low setting with 33% output microwave energy was used.

Analytical Methods

1. Elemental Analysis

The elemental composition (CHN) of palm fiber was examined using Leco CHN-900/CHNS-932/VTF-900 elemental analyser (USA). The instrument determines the elemental composition; Carbon (C), Hydrogen (H), Nitrogen (N) of a sample using a combustion process to break down substances into simple compounds which are then quantified and measured.

2. Composition changes

After the elemental analysis, each sample was analyzed for composition, to determine the cellulose, hemicellulose, and lignin content before and after pretreatment. Standard procedure given by NREL LAP ‘‘Determination of Structural Carbohydrates and Lignin in Biomass” (Sluiter et al., 2012) was used, and the sugar components were analyzed by HPLC. The HPLC system used was Agilent Technologies 11 Series HPLC with G1379A Degasser, G1311A Quatpump, G1315B DAD, G1362A RID, G1328B Man. Inj. column selected was Agilent HiPlex pb with RI detection system. The isocratic mobile phase was deionized water at a flow rate of 0.6ml/min and 80°C. The standard solution 0.1% of lignin was prepared and 20 µL was injected to ascertain the retention time parameters at desired 80 oC temperature level. The complete separation of the component took time from 11-26 min. on the time scale from 0-35 min. The real sample was subjected to HPLC separation and same results obtained thereby confirming the separation of lignin part. The cellulose content of the biomass is enriched with sugars of which glucose is important. The separation of glucose was achieved at retention time of 14 min. when 20 µL of 0.1% standard solution was subjected to analysis. Cellulose comprises of polysaccharide compounds hence many fractions were observed when biomass palm fiber solution was injected. The total time for separation of cellulose part took 50 min. The hemicellulose content of the biomass palm fiber was separated using 20 µL of standard 0.5% xylan solution at retention time of 14-16 min. with overall separation time of 25 min. The separation of the contents carried many sugars but during the process many unknown peaks were observed possibly due to presence of undesired components in the matrices as impurities. The moisture content was obtained by drying overnight in an oven at 105°C following the procedure specified in (NREL) laboratory analytical procedure (LAP) ‘‘Determination of Total Solids in Biomass” (Sluiter et al., 2008a), while the ash content was analyzed by using a benchtop muffle furnace in accordance with NREL LAP ‘‘Determination of Ash in Biomass” (Sluiter et al., 2008b).

RESULTS AND DISCUSSION

The untreated and treated EFB palm fiber were subjected to examination just after their laboratory

Table 1- Elemental Composition of Palm Fiber before and after Pretreatment sample solution preparations. In case of biomass waste it is customary to check the elemental composition of contents present in the matrix. Main components of EFB palm fiber are cellulose and hemicellulose; mainly composed of skeleton carbon, whereas the lignin part contains carbon and nitrogen.

Serial No.	Sample	Carbon %	Hydrogen %	Nitrogen %	C/N ratio
01.	Untreated Palm Fiber	36.07	7.312	2.37	15.2
02.	2% NaOH MW treated Palm Fiber	43.57	6.269	1.899	22.9
03.	3.5% NaOH MW treated Palm Fiber	38.65	5.901	1.136	34.0
04.	5% NaOH MW treated Palm Fiber	38.52	6.975	1.534	25.1
05.	2% Na_2CO_3 MW treated Palm Fiber	30.21	3.896	1.632	18.5
06.	3.5% Na_2CO_3 MW treated Palm Fiber	26.08	2.674	1.318	19.7
07.	5% Na_2CO_3 MW treated Palm Fiber	26.51	3.348	1.481	17.9
08.	2% H_2O_2 MW treated Palm Fiber	21.97	2.313	1.197	18.4
09.	3.5% H_2O_2 MW treated Palm Fiber	31.38	5.436	1.157	27.1
10.	5% H_2O_2 MW treated Palm Fiber	29.41	0.286	1.113	26.4
11.	2% H_2SO_4 MW treated Palm Fiber	29.54	4.027	1.487	19.9
12.	3.5% H_2SO_4 MW treated Palm Fiber	25.97	3.373	1.485	17.4
13.	5% H_2SO_4 MW treated Palm Fiber	24.47	3.515	1.397	17.5

The elemental composition results obtained for one untreated and 12 prepared and microwave assisted pretreated EFB palm fiber suspensions are shown in Table. 1. The untreated sample has a C/N ratio of 15.2 presumably due to presence of high amount of nitrogen in lignin and hemicellulose part of EFB palm fiber with respect to carbon contents. Pretreatment with microwave using NaOH, Na2CO3, H2O2 and H2SO4 with 2%, 3.5% and 5% concentrations gave C/N ratios in the range of (for 2%) 22.9, 18.5, 18.4 and 19.9 respectively, (for 3.5%) 34.0, 19.7, 27.1 and 17.4 respectively and (for 5%) 25.1, 17.9, 26.4 and 17.5 respectively. It is clear from the data that H2O2 with 2% concentration gave the lowest carbon content (21.97%) in comparison to NaOH with 2% concentration gave the highest carbon content (43.57%) (Table.1). From these early findings, it can be concluded that NaOH with 3.5% concentration is found to be the best chemical when pretreated with microwave technique for this analysis where it gives the highest carbon to nitrogen ratio (34.0).

Table 1 shows elemental composition with C/N ratios obtained by microwave assisted chemical pretreatment methods. C/N ratio of 15.2 was observed for untreated palm fiber, while the treated palm fiber gave a range of C/N ratios as lowest as 17.4 for 3.5% sulphuric acid to as high as 34.0 from 3.5% sodium hydroxide. C/N ratio of 25 to 35 is considered to be suitable for optimum biogas production using anaerobic digestion (Mussoline et al., 2013). With respect to changing C/N ratio of palm fiber to optimum level, it was observed that the pretreatments followed the trend sodium hydroxide > hydrogen peroxide > sulphuric acid > sodium carbonate.

Table 2 presents the composition of EFB palm fiber based on cellulose, hemicellulose, lignin, ash and moisture content. All microwave assisted chemical pretreatment methods studied were able to reduce the lignin content of palm fiber. The graphic trend of Fig. 2 shows the percentage decrease of lignin in each pretreatment in comparison to the lignin content of untreated palm fiber (27.32%). Which was degraded to various levels by pretreatment, as 3.5% sodium hydroxide pretreatment was found to be the most effective amongst all pretreatment methods which reduced lignin content to 12.29%. The maximum to minimum lignin content found was in the range of 24.81% for that of sulphuric acid (5%) to 12.29% for sodium hydroxide (3.5%). The trend observed with respect to pretreatments of EFB palm fiber samples for lignin contents was sodium hydroxide < hydrogen peroxide < sodium carbonate < sulphuric acid. Likewise in case of cellulose contents 3.5% sodium hydroxide gave the maximum response (56.69%) against minimum response obtained in 2% sulphuric acid (41.51%). The cellulose content of untreated palm fiber was 35.41%. The ash content was recorded from 5.44% for sodium hydroxide (3.5%) to 8.73% for sulphuric acid (2%) respectively against 6.51% ash contents for untreated EFB palm fiber sample; whereas moisture content was 6.10% for sodium hydroxide (3.5%) to 7.90% for sulphuric acid (3.5%) against 6.90% moisture content for untreated EFB palm fiber sample.

Table 2- The Composition of Palm Fiber before and after Pretreatment

Serial No.	Sample	Cellulose %	Hemicellulose %	Lignin %	Ash %	Moisture %
01.	Untreated Palm Fiber	35.41	19.87	27.32	6.50	6.9
02.	2% NaOH MW treated Palm Fiber	47.11	17.36	19.39	5.74	6.4
03.	3.5% NaOH MW treated Palm Fiber	56.69	15.48	12.29	5.44	6.1
04.	5% NaOH MW treated Palm Fiber	49.34	16.42	15.41	8.13	6.7
05.	2% H_2SO_4 MW treated Palm Fiber	41.51	17.46	22.11	8.73	6.2
06.	3.5% H_2SO_4 MW treated Palm Fiber	44.36	14.54	20.71	8.49	7.9
07.	5% H_2SO_4 MW treated Palm Fiber	45.23	10.47	24.81	8.30	7.2
08.	2% Na_2CO_3 MW treated Palm Fiber	44.38	17.36	20.26	6.60	7.4
09.	3.5% Na_2CO_3 MW treated Palm Fiber	43.49	16.73	23.63	5.45	6.7
10.	5% Na_2CO_3 MW treated Palm Fiber	45.71	16.67	21.34	5.78	6.5
11.	2% H_2O_2 MW treated Palm Fiber	47.66	18.68	15.90	7.36	6.4
12.	3.5% H_2O_2 MW treated Palm Fiber	49.7	14.73	16.91	8.46	6.2
13.	5% H_2O_2 MW treated Palm Fiber	51.28	15.81	14.95	6.76	7.2

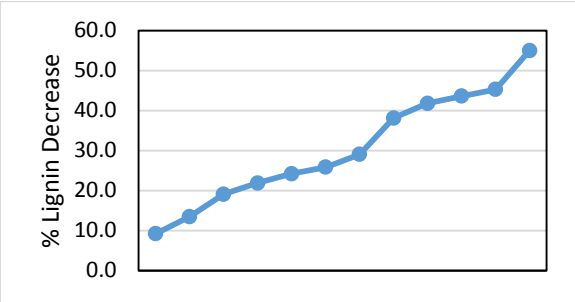

Figure 2- Percentage Reduction of Palm Fiber Lignin after Microwave Pretreatment

Fig. 3 shows the percentage increase of cellulose in each pretreatment in comparison to the cellulose content of untreated palm fiber (35.41%). It shows cellulose increment to different levels by the pretreatments. However a significant percent increment in cellulose was observed in 3.5% sodium hydroxide pretreated palm fiber (56.69%), followed by 5% sodium hydroxide (51.28%), and 3.5% sulphuric acid (49.7%). The percentage reduction of hemicellulose by each pretreatment is shown in Fig.4.

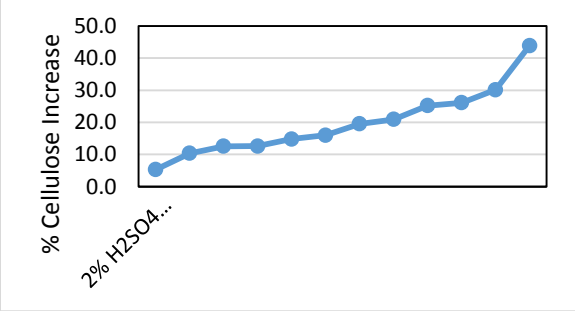

Figure 3- Percentage Increase of Palm Fiber Cellulose after Microwave Pretreatment

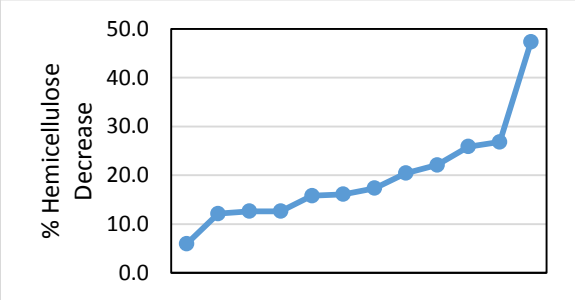

Figure 4- Percentage Decrease of Palm Fiber Hemicellulose after Microwave Pretreatment

Decrease in lignin content causes increase in mainly cellulose content. Cellulose has a very high potential of energy, and has been successfully used for biofuel generation as ethanol or biogas. It has been reported that 100 grams of cellulose can produce upto 51.4 grams of ethanol and 48.6 grams of CO2 (Badger,

2002). It has also been reported that theoretically, 710 ml of biogas can be recovered for each gram of cellulose, with 51 to 56% methane (Khan et al., 1979).

CONCLUSION

The data analysis has shown that the pretreatment performed was effective in changing the chemical composition and behavior of EFB palm fiber samples. The results showed that microwave assisted chemical pretreatment using sodium hydroxide, hydrogen peroxide, sulphuric acid and sodium carbonate with concentrations of 2%, 3.5% and 5% were effective in reducing the lignin content from the untreated palm fiber sample (lignin 27.32%). Sodium hydroxide with 3.5% concentration reduced lignin content significantly to 12.29% while changing the C/N ratio to a desired level (34.0). Thus microwave assisted chemical pretreatment of EFB palm fiber using 3.5% NaOH was considered most suitable.

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