

Microwave-Alkaline Assisted Pretreatment of Banana Trunk for Bioethanol Production

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Abstract: Pretreatment is one of the most important steps in the production bioethanol from lignocellulose materials. Alkaline pretreatment is a common mean of pretreatment but microwave oven could assist its efficiency as it can reduce the pretreatment time and improve the enzymatic activity during hydrolysis. The aim of this paper is to determine lignin removal from banana trunk using microwave-assisted alkaline (NaOH and NH₄OH) pretreatments. The best pretreatment conditions were used for mass pretreatment before hydrolysis and fermentation. The result shows that, optimum lignin removal was with microwave-assisted NaOH pretreatment with the removal of up to 98% lignin at 2% (w/v (weight/volum)) sodium hydroxide, 170 W microwave power at 10 min. Microwave-assisted ammonium hydroxide pretreatment achieved 97% lignin removal at 1% ammonium hydroxide concentration and 680 W microwave power at 5 min. Microwave- alkaline assisted pretreatment increased the yield and quality of fermentable sugar after enzyme hydrolysis with NH₄OH and ammonium hydroxide yielding 40% and 39% of ethanol, respectively. This result reveals that, well controlled microwave- alkaline assisted pretreatment of banana trunk could effectively remove lignin and give high bioethanol yield.

Key words: Microwave, alkali, pretreatment, cellulosic, banana trunk, bioethanol.

1. Introduction

Bioethanol or ethyl alcohol (C₂H₅OH) is a colourless gaseous or liquid fuel derived from hydrolysis and fermentation of glucose from cellulose biomass. It is produced from distillation of ethanolic waste gotten from fermentation of cellulosic biomass [1]. Bioethanol can be produced from biomasses containing cellulose such as agricultural waste, woody materials, by-product of organic materials, forest residues, herbaceous material, municipal waste, etc. [2]. Alternative liquid fuels from various sources have been sought for many years, and since the cost of raw materials which can account up to 50% of the total production, cost is one of the most significant factors to affect the economy of alcohol, nowadays, efforts are more concentrated on using cheap and abundant raw

materials [3]. No other sustainable option for production of transportation fuels can match ethanol made from lignocelluloses biomass with respect to its dramatic environmental, economic and infrastructure advantages [4]. The lignocellulosic materials include agricultural residues, MSW (municipal solid wastes), pulp mill refuse, switch grass and lawn, garden wastes [5, 6]. The structures of lignocellulose material are rigid and recalcitrant. These characteristics provide plant it mechanical support and prevent it from chemical degradation and enzymatic hydrolysis for bioethanol production [7]. Pretreatment of lignocellulose biomass for bioethanol production is an important process step done to remove lignin and hemicellulose from biomass so that the residual cellulose can be hydrolyzed to fermentable sugar [8]. Acid pretreatment of biomass result in disruption of covalent bond, hydrogen bond and Vander Waals forces holding the constituent of the biomass together therefore resulting in the solubilisation and

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decrystallization of biomass [9]. Alkali pretreatment on the other hand causes swelling of biomass through saponification reaction [10]. Alkali pretreatment disrupts the structure of lignin and breaks the bond between lignin and other cellulose component of biomass thereby increasing their susceptibility to enzymatic hydrolysis [8]. Enzymatic hydrolysis involves mixture of enzymes such as cellulose and hemicellulases [11]. These enzymes must be able to break down lignocellulosic materials and convert it to fermentable glucose [12]. Enzymatic hydrolysis has low utility lost compare to acid hydrolysis and environmental friendly [13]. Banana truck is a promising cellulose and lignocellulosic plant which generates a significant amount of waste, approximately 200 t/ha/year [14]. Despite its cellulose (63%-64%) and hemicellulose (19%) content [15], it is not often used as a raw material for bioethanol production probably due to its lignin content (15.07%). The aim of this study is to determine lignin removal of banana trunk using alkaline pretreatment (NaOH and NH₄OH) microwave assisted alkaline pretreatment. The effectiveness of these pretreatments was evaluated by bioethanol yield through fermentation.

2. Materials and Methods

Materials used are of analytical grades unless otherwise stated. Methods followed the stages and steps as outlined.

2.1 Collection of Samples

Banana trunk was collected from Bosso, Minna, and was dried in an oven and grinded to powder. The sample was further sieved into smaller particles and kept for further analysis.

2.2 Chemicals

Sodium hydroxide (NaOH), ammonium hydroxide (NH₄OH) and distilled water.

2.3 Chemical Pretreatment

Banana trunk biomass was subjected to pretreatment

with sodium hydroxide (NaOH) of 2%, 4%, 6%, 8% and 10% concentration in the ratio 10:1, and then placed in the water-bath with temperature of 60, 70, 80, 90 and 100 °C for 1 h, banana trunk was also immersed in ammonium hydroxide (NH₄OH) with the concentration of 1%, 2%, 3%, 4%, 5% and also placed in the water-bath for 1 h with the temperature ranging from 60 °C to 100 °C. The mixture was then filtered with muslin cloth and the residue was washed with clean water until the pH was neutral and dried in an oven to remove excess water and moisture and then kept for further analysis.

2.4 MAA (Microwave Assisted Alkaline) Pretreatment

Banana trunk powder was immersed in 2%, 4%, 6%, 8% and 10% NaOH solution in the ratio of 10:1 of liquid to solid (v/w (volum/weight)). The mixture was then placed in a microwave oven 5, 10, 15, 20 and 25 min at the varying power of 170, 340, 510, 680 and 850 W. After this process, the mixture was then filtered and the residue was washed with clean water until the pH was neutral and the dried in the oven and kept afterwards for further analysis. Also the sample was immersed in ammonium hydroxide (NH₄OH) with the varying concentration of 1%, 2%, 3%, 4% and 5% in the ratio of 10:1 and was then placed in a microwave oven for 5, 10, 15, 20 and 25 min at varying power of microwave oven at 170, 340, 510, 680 and 850 W. The sample was then filtered and the residue was washed with clean water until the pH was neutral and afterwards dried in the oven to remove water and moisture. The dried residue was then kept for further analysis. Optimum condition obtained from both microwave-assisted alkaline pretreatments were used generate bulk pretreated banana trunk biomass. This biomass was thereafter hydrolyzed with cellulose and fermentation to produce bioethanol.

3. Results and Discussion

Lignin removal using ammonium hydroxide (NH₄OH) and sodium hydroxide (NaOH) in water bath

pretreatments are shown in Figs. 1 and 2. The reduction in the weight of the residue is proportional to the lignin removed. The effect of NH₄OH concentration is not significant on lignin removal at 60 °C and 70 °C. The highest removal of legnin was observed with 2% NH₄OH at 100 °C. On the other hand, lowest removal of legnin was found at 100 °C with 4% NH₄OH. Concentration of NaOH had significant influence on lignin removal as shown in Fig. 2. It was found that, when NaOH concentration increased from 2% to 10%, there was general reduction in the weight of residual sample at some of the experimental temperatures (70, 90 and 100 °C). The highest biomass reduction (lignin removal) was observed with 8% NaOH at 80 °C [16],

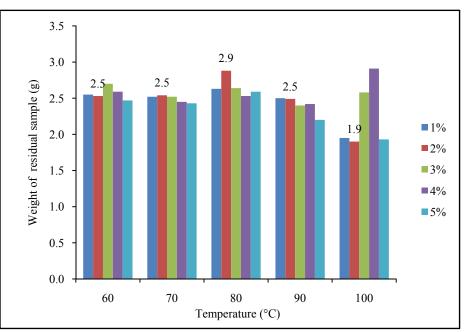


Fig. 1 Mean weight of residual biomass after pretreatment with ammonium hydroxide with the aid of water bath at different temperature.

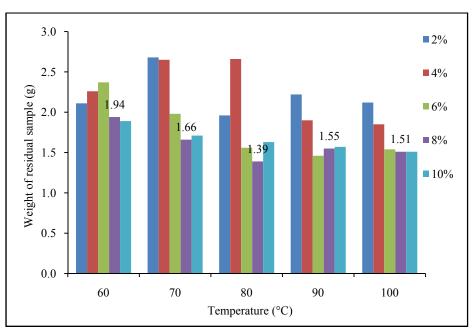


Fig. 2 Mean weight of residual biomass after pretreatment with sodium hydroxide with the aid of water bath at different temperature.

studied the effectiveness of sulfuric acid, sodium hydroxide, hydrogen peroxide and ozone pretreatments for enzymatic conversion of cotton stalks. They found that, 2% sodium hydroxide pretreatment resulted in the highest level of delignification at 121 °C. Earlier studies have shown that, 2% sodium hydroxide, would remove up to 65% lignin in corn stover [17]. Also [18] achieved over 55% reduction in the lignin content within first 0.5 h of pretreatment using 0.5% sodium hydroxide solution at 120 °C. The pretreatment achieved in this study is in correlation with previous studies based on the suggestion of alkaline pretreatment at lower temperature with high alkaline concentration or vice versa [19]. Sodium hydroxide solution has shown to be more effective in deligninfication of banana trunk biomass than aqueous ammonium hydroxide. Sodium hydroxide is one of the most effective alkaline reagents and has been used to treat a variety of lignocellulosic feedstock [20].

3.1 Microwave-Assisted Alkaline Pretreatment

NaOH and NH₄OH are dipolar reagents and the alignment and migration of their charged ions and water molecules in rapidly alternating electromagnetic field results in a friction which generate heat within a short period of time in microwave system. The electromagnetic field of microwave irradiation applies force that changes their direction rapidly at the rate of 2.4×10^9 times per second [21]. These characteristics can increase the process of chemical, biological and physical pretreatment [22].

3.1.1 Graphical Representation of Microwave-Assisted Sodium Hydroxide Pretreatment at Varied Time Using Surfer-11 Model

Generally, microwave assisted pretreatment using different concentrations of ammonium hydroxide yielded the lowest weight delignification at the concentration of 1% and irradiation power of 680 W after time 10 min of pretreatment. This is because extended pretreatment [23] and charring of the sample at very high temperature (850 W) during microwave

assisted alkaline pretreatment [24] is likely to result in loss of carbohydrates to the pretreatment liquor. (Figs. 3-12). However, the present study has shown that, microwave-assisted sodium hydroxide pretreatment at 2% (*w*/*v*) concentration, 170 W for 10 min (Fig. 4) was the best conditions for delignification of banana trunk biomass.

The result shows a rise and fall pattern in weight of sample as the power of irradiation and concentration of sodium hydroxide is increased but shows the lowest reduction at concentration of 8% and power of 680 W.

Microwave assisted pretreatment using different concentration of sodium hydroxide at different power shows an increase delignification as the irradiation power and concentration of NaOH increased. The optimum condition of pretreatment was observed at 2% NaOH and 170 W irradiation power. This optimum condition gave the overall highest delignification of banana trunk cellulose.

The pretreatment condition at 15 min shows a rise and fall pattern as the irradiation power and concentration of NaOH was increased, and the lowest weight of residual cellulose was 1.26 and was observed as the concentration and power were increased.

Pretreatment condition at 20 min shows a rise and fall pattern in the weight of residual sample. Increase in the irradiation power and concentration reduces the weight of the residual sample.

Microwave assisted pretreatment using different irradiation power and concentrations of sodium hydroxide shows a rise and fall pattern in the weight of residual cellulose, and the optimum condition was observed at 2% NaOH and 170 W irradiation power.

3.1.2 Graphical Representation of Microwave Assisted Ammonium Hydroxide Pretreatment Using Surfer-11 Model

Microwave assisted pretreatment using ammonium hydroxide of different concentration and irradiation power shows a rise and fall pattern of residual weight but gives a sharp fall at 680 W of irradiation power.

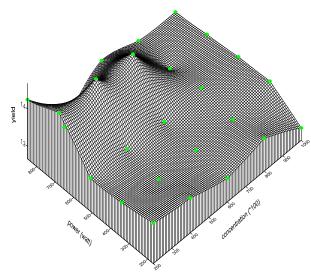


Fig. 3 Microwave assisted pretreatment using sodium hydroxide for 5 min.

Regression coefficient Z (X, Y) = A00 + A01 (Y) + A10 (X);

A00 = 1.3592;

A01 = $7.764705882363 \times 10^{-0.05}$;

after pretreatment for 5 min.

A10 = -0.00136;

Coefficient of multiple determination $(R^2) = 0.210322865663$; Surface definition = simple planar surface;

X = Concentration, Y = Power, Z = Weight of residual sample

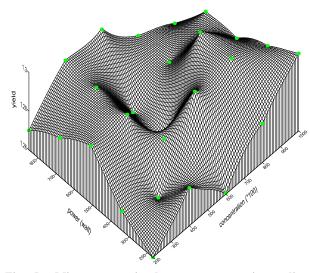


Fig. 5 Microwave assisted pretreatment using sodium hydroxide for 15 min.

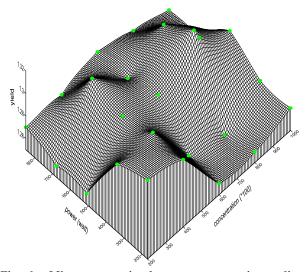
Regression coefficient Z (X, Y) = A00 + A01 (Y) + A10 (X); A00 = 1.2762;

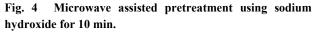
 $A01 = -4.7058823529407 \times 10^{-0.06};$

 $A10 = -9 \times 10^{-0.06};$

Coefficient of multiple regression $(R^2) = 0.0389245585875$; Surface definition = simple planar surface;

X = Concentration, Y = Power, Z = weight of residual sample after pretreatment for 15 min.





Regression coefficient Z (X, Y) = A00 + A01 (Y) + A10 (X);

A00 = 1.2756;

 $A01 = 2.4706882352943 \times 10^{-0.05};$

A10 = $-9 \times 10^{-0.05}$;

Coefficient of multiple regression $(R^2) = 0.0436746987952$; Surface definition = simple planar surface;

X = Concentration, Y = Power, Z = Weight of residual sample after pretreatment for 10 min.

Fig. 6 Microwave assisted pretreatment using sodium hydroxide for 20 min.

Regression coefficient Z (X, Y) = A00 + A01 (Y) + A10 (X);

A00 = 1.3038;

 $A01 = -1.5294117647057 \times 10^{-0.05};$

 $A10 = -2.4 \times 10^{-0.05};$

Coefficient of multiple regression (R^2) = 0.124832439678;

Surface definition = simple planar surface;

X = Concentration, Y = Power, Z = weight of residual sample after pretreatment for 20 min.

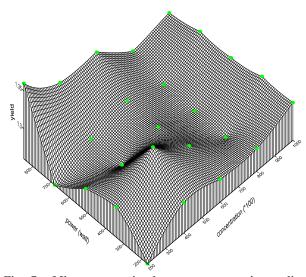


Fig. 7 Microwave assisted pretreatment using sodium hydroxide for 25 min.

Regression coefficient Z (X, Y) = A00 + A01 (Y) + A10 (X);

A00 = 1.2912;

 $A01 = 1.4117647058825 \times 10^{-0.06};$

A10 = -6.000000000018 \times 10^{-0.06};

Coefficient of multiple regression $(R^2) = 0.02722323049;$

Surface definition = simple planar surface;

X = Concentration, Y = Power, Z = weight of residual sample after pretreatment for 25 min.

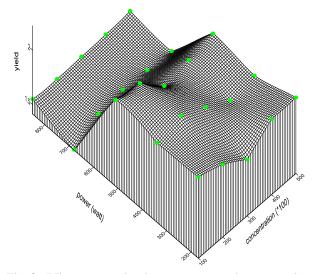


Fig. 8 Microwave assisted pretreatment using ammonium hydroxide for 5 min.

Regression coefficient Z (X, Y) = A00 + A01 (Y) + A10 (X); A00 = 2.3644;

A01 = -0.00093176470688205;

A10 = -0.000614;

Coefficient of multiple regression $(R^2) = 0.68979218801$; Surface definition = simple planar surface;

X = Power, Y = Concentration, Z = weight of residual sample after pretreatment for 5 min.

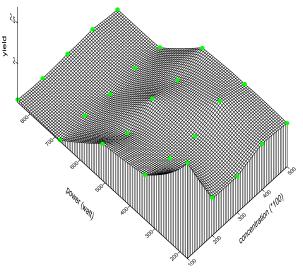


Fig. 9 Microwave assisted pretreatment using ammonium hydroxide for 10 min.

Regression coefficient Z (X, Y) = A00 + A01 (Y) + A10 (X);

A00 = 2.4112; A01 = 0.001001764705882;

A10 = 0.000162;

410 = 0.000102,

Coefficient of multiple regression $(R^2) = 0.72741588692;$

Surface definition = simple planar surface;

X = Power, Y = Concentration, Z = weight of residual sample after pretreatment for 10 min.

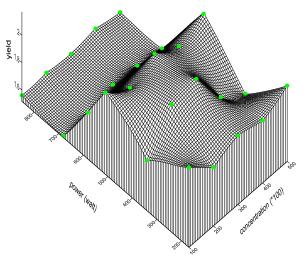


Fig. 10 Microwave assisted pretreatment using ammonium hydroxide for 15 min.

Regression coefficient Z (X, Y) = A00 + A01 (Y) + A10 (X);

A00 = 2.668;

A01 = -0.00078;

A10 = -0.000134;

Coefficient of multiple regression (R^2) = 0.621727953942;

Surface definition = simple planar surface;

X = Power, Y = Concentration, Z = weight of residual sample after pretreatment for 15 min.

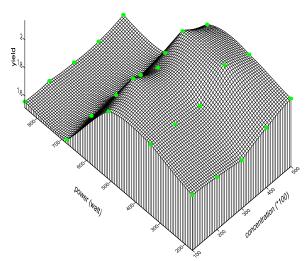


Fig. 11 Microwave assisted pretreatment using ammonium hydroxide for 20 min.

Regression coefficient Z (X, Y) = A00 + A01 (Y) + A10 (X); A00 = 2.156;

A01 = -0.00071647058823529;

A10 = 0.000114;

Coefficient of multiple regression $(R^2) = 0.572491400311;$

Surface definition = simple planar surface;

X = Power, Y = Concentration, Z = weight of residual sample after pretreatment for 20 min.

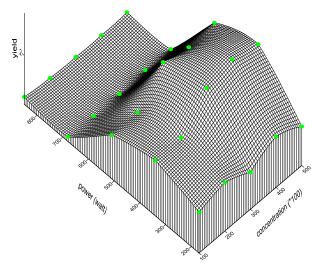


Fig. 12 Microwave assisted pretreatment using ammonium hydroxide for 25 min.

Regression coefficient Z (X, Y) = A00 + A01 (Y) + A10 (X); A00 = 2.234;

A01 = -0.00087882352941177;

A10 = 0.000318;

Coefficient of multiple regression $(R^2) = 0.508299550688$; Surface definition = simple planar surface;

X = Power, Y = Concentration, Z = weight of residual sample after pretreatment for 25 min.

Pretreatment condition for 10 min gives a gradual reduction in weight of residual sample and a sharp decrease when the irradiation power was increased to 680 W. The optimum condition yielding 1.53 g was observed at concentration of 1% NH₄OH at irradiation power 680 W.

Pretreatment condition after 15 min shows a rise and fall pattern in the weight of residual cellulose and a sharp reduction was observed at 680 W. The optimum conditions of pretreatment after 15 min were observed as the concentration and power was increased at 3% and 5% NH_4OH and power irradiation of 680 W and 850 W, respectively.

Pretreatment condition after 20 min of pretreatment with microwave irradiation shows a gradual fall in the weight of residual cellulose and a sharp reduction at 680 W. Increase in concentration of NH₄OH on the other hand produces a rise and fall pattern in the weight of residual cellulose.

Pretreatment condition after 25 min shows a gradual rise and fall in the weight of residual cellulose and a sharp decrease when power was increased to 680 W. The optimum yield was observed at 680 W and 850 W.

This implies that, low power, short time and low concentration of sodium hydroxide were required for high removal of lignin when using microwave radiation. The present observation agrees with that of [25] who observed that, the pretreatment of Bermuda grass with microwave assisted sodium hydroxide pretreatment yielded optimum result at the irradiation power of 250 W, 1% NaOH concentration for 10 min which removed 65% of lignin while retaining 87% of the glucan. Furthermore, the weight of residual sample reduction in microwave-assisted sodium hydroxide pretreatment (Fig. 4) was higher than that of NaOH pretreatment alone (Fig. 2). This observation supports the finding of [26] who reported that, during alkali pretreatment of wheat straw, with microwave assisted heating, lower sugars losses and higher hydrolysis rates were observed than that of conventional alkaline pretreatment alone.

Microwave assisted ammonium hydroxide pretreatment showed lowest delignification at 1% (w/v)concentration, 680 W under 10 min (Fig. 9) indicating that, high power, short time and low concentration of alkali were required for high removal of lignin. Although microwave-assisted ammonium hydroxide pretreatment yielded better delignification of banana trunk biomass at lower alkaline concentration than microwave-assisted NaOH pretreatment, solution microwave-assisted ammonium was considered as less efficient due to the tendency of reduced sugar yields during hydrolysis when using high power microwave assisted alkaline pretreatment [24]. However, microwave assisted ammonium hydroxide pretreatment was prefer over the convention ammonium hydroxide pretreatment alone (Fig. 1) as a result of the shorter time (10 min) and lower ammonium concentration (1%) required. It is therefore clear from this study that, microwave assisted pretreatment achieves better delignification of banana trunk biomass than pretreatment with sodium hydroxide and ammonium hydroxide alone.

Enzymatic hydrolysis and fermentation of pretreated biomass obtained from the optimum conditions of microwave assisted sodium hydroxide pretreatment yields 300 mL of 40% bioethanol, while microwave-assisted ammonium hydroxide pretreatment yields 150 mL of 39% bioethanol. Many reports have shown that, short pretreatment duration when using microwave radiation enhances the destruction of crystalline arrangement of starch in their hydrolysis to reducing sugar [27].

4. Conclusions

The result obtained from this study shows that, dilute sodium hydroxide (NaOH) of 2% at 170 W with microwave irradiation exposure of 10 min yields the highest amount of reducing sugar for bioethanol production. Therefore, dilute alkaline pretreatment when combined with microwave irradiation is a promising method for lignin removal from lignocellulose biomass, although more researches are still focused on how to improve this method for a better yield. The use of dilute sodium hydroxide at low irradiation power for microwave assisted alkaline pretreatment is an effective way of removing lignin from lignocellulose biomass under short period of time, and hence gives a higher turnover rate of bioethanol production.

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