Experimental and Kinetic Modelling Studies on the Acid-Hydrolysis of Banyan Wood Cellulose to Glucose

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Abstract

Bioconversion of agricultural waste products to produce value-added fuels and chemicals offers potential economical, environmental and strategic advantages over traditional fossil-based products. The kinetics of acid hydrolysis of cellulose isolated from banyan wood sawdust was studied at sulphuric acid concentration of 2 to 5 M (moles/dm³) and temperature ranging between 40 - 80 °C in a stirred conical flask which served as a batch reactor. The results showed that the rate of acid hydrolysis by virtue of glucose yield generally increased with increase in acid concentration and temperature used. The experimental data were fitted to integrated first order rate kinetics and the results obtained suggested a first order rate of glucose formation from banyan wood cellulose. The specific rate constant relatively increased with increase in acid concentration energy estimated from Arrhenius equation was found to be 6.75 kJ/mole. This value suggests the ease with which hydrolysis can occur between the four agricultural wastes cellulose.

Keywords: Acid-hydrolysis; Arrhenius equation; First-order kinetics; Glucose; Wood cellulose.

1. Introduction

The agricultural activities of man have resulted in the production of large quantities of agricultural waste biomass that tends to dominate and pollute the environment. Many of these agro-wastes are allowed to rot away not utilized (Obot et al., 2008). These wastes biomass consist of cellulose, hemicelluloses lignin and other materials called extractive (Ghose1956, Aberuagba, 1997). Among all the constituents of agricultural wastes biomass, cellulose constitutes the highest percentage because it is a strong elastic material that forms the cell wall of nearly all plants (Aberuagba, 1997). The cellulose can be hydrolyzed to produce glucose for human needs, which can further be used as substrates for fermentative production of useful products like alcohols (John et al., 2007; Benkun et al., 2009). Bioconversion of agricultural wastes biomass to produce value-added fuels and chemicals offers potential economical, environmental and strategic advantages over traditional fossil-based products (Anex et al., 2007). Generally, agricultural wastes from different sources have different physical properties such as surface area, lignifications, crystallinity and other different chemical compositions that could hinder the accessibility susceptibility of cellulose for hydrolysis (Aberuagba, 1997; Caritas and Humphrey, 2006). However, they may be modified to enhance their susceptibility to hydrolysis through pretreatment processes. The pretreatment process (physical, chemical and/or microbial) alters the structure and compositions of the agricultural wastes biomass and this removes extractives, lignin and hemi-cellulose, reduce cellulose crystallinity and increase porosity (Aberuagba, 1997; Ander and Ericson, 1983; Sun and Cheng, 2002). Over the last decades, the hydrolysis of cellulose and lignocellulosic materials has been a subject of intensive research for the development of large scale conversion processes that would be of benefit to mankind (Gamez et al., 2004; Hahn-Hagerdal et al., 2006; Qu et al., 2006; Girisuta et al., 2008; Sarkar and Aikat, 2012; Ajani et al., 2012; Kupiainen et al., 2014). These processes would among other things help to solve modern disposal problems, reduce pollution of the environment and reduce man's dependence on fossil fuels by providing a convenient and renewable source of energy in the form of bioethanol (Cowling et al., 1976).

Conversion of cellulose and lignocellulosic biomass to glucose and other monomeric sugars can be achieved by acid and enzyme hydrolysis (Badger, 2002; Benkun, et al, 2009, Megawati et al., 2010; Wu et al., 2010). The relative advantages of enzyme and acid hydrolysis of cellulose is a subject of continuing research study. The enzymatic process is believed to be the most promising technology because enzymatic hydrolysis is milder and more specific and does not produce by products (Wen et al., 2004; Benkun et al, 2009). However, enzymatic hydrolysis of cellulose have been observed not to be economically viable because of high cost of enzymes, slow rate of depolymerization and high enzyme loading to realize reasonable rates and yields (Layokun, 1981; Aberuagba, 19997; Grohmann and Baldwin, 1995; Wyman, 1999). The advantages of acid hydrolysis for peel liquefaction and releasing carbohydrates prior to enzymatic treatment have been studied (Vaccarino et al; 1989; Grohmann et al., 1995; Talebnia et al., 2008). Acid hydrolysis of cellulosic biomass is relatively fast and of low cost (Palmqvist and Hagerdal, 2000; Megawati et al., 2010). The key variables that might have impacts on the rate and extent of cellulose and lignocellulosic biomass by acid hydrolysis are temperature, acid concentration (or pH), total solid fraction (TS) and time duration (Grohmann, et al., 1995;

Talebnia et al., 2008). Several studies have been made on cellulose hydrolysis using purified raw materials especially commercial cellulose as well as cellulose derived from agricultural wastes such as *Eucalyptus* wood, *Meranti* wood sawdust, corncobs, sugarcane bagasse, sorghum straw, brewer's spent grain, and OPEFB using dilute sulphuric acid and very high temperature ($100 - 250^{\circ}$ C) (Parajo et al., 1994; Dominquez et al., 1997; Lavarack et al., 2002;Mussato and Roberto, 2005; Rahman et al., 2007); however, with very few studies using concentrated sulphuric acid and moderate temperature have been carried out (Aberuagba, 1997; Ajani et al., 2012).

Wide varieties of biomass sources are available for further conversion and utilization. Proper selection of the biomass feedstock is of paramount importance from both a techno- and socio-economical point of view. The biomass feedstock should not compete with the food chain and waste streams with a low or even negative value, such as agricultural waste, are preferred. Furthermore, it is also advantageous to select sources that are not prone to diseases and are preferably available throughout the year (Girisuta et al., 2008). Based on these criteria, the wood sawdust could be an excellent biomass feedstock for further conversion and utilization. Wood sawdust is a lignocellulosic byproduct of sawmill that is available at low cost throughout the year (Islam and Mimi Sakinah, 2011). It is produced in enormous quantities by sawmills and the economical disposal of them is a serious problem to the wood based industries. Sawdust is commonly used as fuel in producing plants and local utilities. Other uses of sawdust are: as litter and bedding material in poultry and livestock structures, for the production of fiberboards and paper pulp (Harkins, 1969; Arends et al., 1985; Islam and Mimi Sakinah, 2011). The hydrolysis of *Banyan* wood sawdust (BWS) to produce glucose solution could be a good alternative use for this abundant resource. The objective of this study is to examine the effects of concentrated sulphuric acid concentration and low to moderate temperature on the hydrolysis of cellulose obtained from *Banyan* wood sawdust and to evaluate their kinetics.

2. Materials and Methods

Wood (Banyan species) was obtained from farmers in Ogbomoso, Nigeria. Sulfuric acid (specific gravity 1.8; 98% purity) used for hydrolysis and Diethyl ether used for the removal of lignin and extractives which are products of E. Merck (Darmstadt Germany) were purchased from a chemical store in Ibadan, Nigeria. Other chemicals used were of analytical or biochemical grade.

2.1 Pretreatment of Banyan wood and Cellulose Isolation

Samples of banyan wood were oven dried at 70 °C until a constant weight was obtained and further dried in a furnace for 10 min. The dried wood samples were reduced to very small sized particles of about 300 µm by grinding using a grinding machine (Flammer Mill, Model 200, UK) incorporated with 2 mm sieve. Cellulose was isolated from the small particle sized banyan wood samples using the modified procedure described by Layokun (1981). Diethyl ether (100 ml) was added to the sample (300 g) in a 250 ml Erlenmeyer conical flask so as to remove the extractives. The resultant residue (free of extractives) was filtered and washed thoroughly with sterile distilled water. To the washed residue was added 500 ml of 14M sulfuric acid which then dissolved the cellulose and hemi cellulose leaving lignin as a hard precipitate. Lignin was filtered off and 8M sodium hydroxide solution was added to the filtrate to obtain a residue that was predominantly cellulose, while hemi cellulose remained in solution. The solution was filtered and the resultant cellulose residue was then washed thoroughly with sterile distilled water until a neutral pH was obtained. The cellulose residue was oven dried at 80 °C until a constant weight was obtained for subsequent hydrolysis.

2.2 Experimental Design for Acid Hydrolysis of Cellulose

To 30 g sample of the cellulose in a 250 ml conical flask which served as a batch reactor was added 100 ml of concentrated sulfuric acid (2 moles/dm³). The flask was placed in a gyratory shaker set at a temperature of 40 $^{\circ}$ C with an agitation speed of 150 rpm and was allowed to operate for 3 h; and at intervals of 30 min, samples were withdrawn to determine the glucose concentration. The experiment was repeated at other temperatures of 50, 60, 70 80 $^{\circ}$ C and sulfuric acid concentrations of 4, 4.5 and 5 moles/dm³, respectively.

2.3 Determination of Glucose Concentration

The reducing sugar content (glucose) was determined by the DNS method with glucose as standard (Miller, 1959; Marsden et al., 1982). Absorbance was measured at 540 nm. However, the DNS reagent was modified according to Mwesigye (1988). Two hundred grams of potassium sodium tartarate (Rochelle salt) were dissolved in 200 ml of sterile distilled water. Ten grams of sodium hydroxide was measured at 540 nm. However, the DNS reagent was modified according to Mwesigye (1988). Two hundred grams of potassium sodium tartarate (Rochelle salt) were dissolved in 200 ml of sterile distilled water. Ten grams of sodium hydroxide was measured at 540 nm. However, the DNS reagent was modified according to Mwesigye (1988). Two hundred grams of potassium sodium tartarate (Rochelle salt) were dissolved in 200 ml of sterile distilled water. Ten grams of sodium hydroxide was also dissolved separately in 200 ml of sterile distilled water in a 500 ml beaker. To the sodium hydroxide solution was added 10 g of DNS (3, 5dinitrosalicyclic acid) and 2.52 ml (2 g) of 80% (w/v) phenol simultaneously. After

stirring to complete dissolution, the mixture was added to the Rochelle salt solution. The resultant solution was then made up to one litre with sterile distilled water. This mixture gave the stock of the modified DNS reagent containing 1% (w/v) DNS acid, 0.2% (w/v) phenol, 1% (w/v) sodium hydroxide and 20% (w/v) Rochelle salt (Mwesigve, 1988). The DNS reagent was then stored under refrigeration in an amber coloured bottle.

2.4 Yield Calculation and Kinetics of Acid Hydrolysis

For molar cellulose concentration (Cc), cellulose is treated as glucan (anhydrousglucose), i.e.

 $MM_{glucanunit} = MM_{glucose} - MM_{H2O}$. In this way, cellulose is converted to glucose equivalents, which represent the maximum theoretical glucose from cellulose. The cellulose concentration as glucose equivalents is calculated as in Eq. (1) (Kupiainen et al., 2014):

$$C_{C} = \frac{W_{C}}{V \times MM_{glucanunit}}$$
(1)

Where W_c is the mass of cellulose or the mass of weighed cake from experiment, V is the liquid volume, and

MM is the molar mass. The glucose yield $\begin{pmatrix} Y_{glu \cos e} \end{pmatrix}$ is defined in this study per initial cellulose concentration as glucose equivalents:

$$Y_{glu\cos e} = 100 \times \left(\frac{C_{glu\cos e}}{C_{C,0}}\right)$$
(2)

Numerous kinetic models for the acid hydrolysis of lignocelluloses are available in the literature. The first model was proposed by Saeman (1945) based on the hydrolysis of cellulose to glucose which is subsequently converted to decomposition products as shown in the following reaction scheme:

Polymer → monomer → decomposition product

The model representing cellulose hydrolysis involves a series of pseudo-homogeneous irreversible first-order reactions. The decomposition reactions were negligible in this study as the operating conditions were not favorable for degradation of glucose, hence the model can be simplified by the following reaction scheme: Cellulose — Glucose

Hence, the cellulose concentration C as a function of time can be expressed as:

$$C_o - X = C_o \exp(kt) \tag{3}$$

(4)

Where C_o , the initial biomass cellulose concentration, k, specific rate constant (min⁻¹), $C_o - X$, biomass cellulose concentration at time t (g/l), X, glucose content (g/l), and t, time (min). On the basis of a first-order reaction for the hydrolysis, Eq. (3) becomes:

$$\ln(\frac{C_o}{C_o - X}) = -kt$$

Thus, equation (2) allows the natural logarithmic plots of experimental values of $C_o/C_o - X$ versus process time (t) in which straight lines obtained are indications of the validity of first-order reaction kinetics for the biomass cellulose acid hydrolysis. The intensity of heat on hydrolysis of biomass cellulose can be described by the Arrhenius model such that temperature dependence of k closely follows the equation:

$$k = k_R \exp\left[-\frac{E_a}{R} \left(\frac{1}{T}\right)t\right]$$
⁽⁵⁾

Where k_R , pre-exponential constant (min⁻¹); E_a , activation energy (KJ/mol); R, ideal gas constant (8.314) J/mol.K); T, actual temperature (K) and t, time (min). The regression of the natural logarithm of the degradation rate constant $\binom{k}{T}$ against $\binom{1}{T}$ in which a straight line curve is obtained indicates that the

Arrhenius model was fulfilled and slope of this curve (Arrhenius plot) is equal to $\sqrt[n]{R}$ and allowed estimation of the activation energy.

3. Results and Discussion

3.1 Effect of operating conditions on glucose yield

The percentage cellulose yield from banyan wood biomass is 28.4%. Benkun et al. (2009) obtained 60.12% cellulose from the acid hydrolysis of wheat straw. Fig. 1 shows the effect of acid concentration on glucose yield from the acid hydrolysis of banyan wood cellulose.

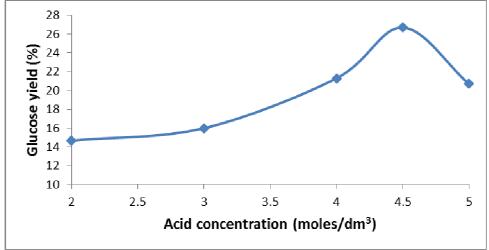


Fig. 1: Effect of sulphuric acid concentration on glucose yield from acid hydrolysis of banyan wood cellulose

The results showed that there is a general increase in glucose yield as the acid concentration increased in the range of 2 M to 5 M (mole/dm³). However, there was a decline in yield from an acid concentration of 4.5 to 5 M (moles/dm³). Similar observations have been reported for the acid hydrolysis of cellulose from maize cobs, groundnut shells (Aberuagba, 1997), banana skin, cowpea shells, maize stalks, rice husk (Ajani et al., 2012); and commercial microcrystalline cellulose (Kupiainen et al., 2014). This observation could be attributed to the fact that at high acid concentration and relatively high temperature; glucose can be converted to organic acid which led to a decrease in glucose concentration (Aberuagba, 1997). This suggests that maximum glucose yield could be obtained at low to moderate acid concentration. Talebnia et al. (2008) reported that at low acid concentration and low temperature, sugar yield increases with increase in dilute acid concentration.

The effect of temperature on glucose yield from acid hydrolysis of banyan wood cellulose is shown in Fig. 2.

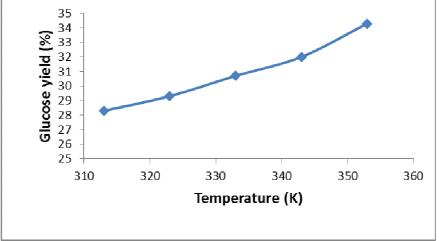


Fig. 2: Effect of temperature on glucose yield from acid hydrolysis of banyan wood cellulose

From Fig 2, it is seen that the glucose yield from acid hydrolysis of banyan wood cellulose increased with increase in temperature. Similar observations have been reported (Aberuagba, 1997; Ajani et al., 2012; Kupiainen et al., 2014). Talebnia et al. (2008) reported that in the acid hydrolysis of orange peels at low temperature range, sugar yield increased with increase in temperature and at very high temperature range, sugar yield declines. However, Megawati et al. (2010) reported that in the acid hydrolysis of rice husk, at high temperature range ($160 - 220^{\circ}$ C), total sugar concentrations increased with increase in temperature. Layokun (1981) has also observed an increase in glucose yield with temperature for acid hydrolysis of saw dust.

3.2 Kinetic modelling

An integrated pseudo first- order rate kinetics (Eq. (3)) was applied to the experimental result data obtained at

different acid concentration and temperature, respectively. The plot of $\ln(\sqrt{C_o - X})$ versus time (t) gave a straight line for each of the considered acid concentration and temperature as shown in Figs. 3 and 4, with the specific rate constant 'k' estimated from the slope.

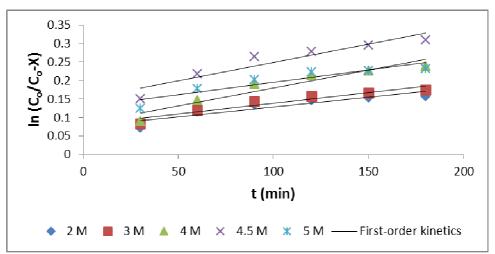


Fig. 3: First-order kinetic model fitted to the experimental data at different sulfuric acid concentration

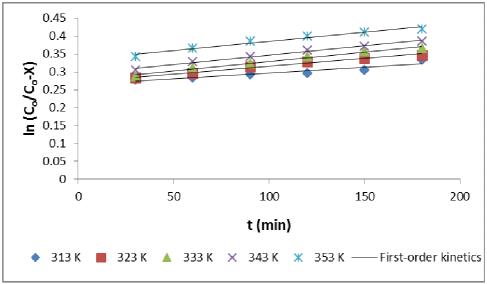


Fig. 4: First-order kinetic model fitted to the experimental data at different hydrolysis temperature The straight line obtained in Figs. 3 and 4 for acid concentration (2 - 5 M) and temperature (40 - 80 °C) investigated with relatively high coefficient of determination (R^2) that ranges from 0.85 to 0.99 suggests a first order rate of glucose formation from banyan wood cellulose hydrolysis by sulfuric acid. The specific kinetic rate constants (k) are presented in Table 1.

Table 1: Values of specific rate constant and coefficient of determination obtained from first-order kinetic model fitted to the acid hydrolysis experimental data for banyan wood cellulose

Operating Condition	Specific rate constant	Coefficient of determination
	$(k) \times 10^{-4} \min^{-1}$	(R^{2})
Sulphuric acid concentration (moles/dm ³)		
2	5.3	0.8469
3	5.8	0.9096
4	9.6	0.9102
4.5	9.9	0.8829
5	6.7	0.8364
Temperature (K)		
313	7.8	0.9889
323	8.3	0.9537
333	9.0	0.9499
343	9.4	0.9423
353	10.6×10^{-4}	0.9504

The results in Table 1 revealed that k generally increases with increased acid concentration and temperature, respectively. The specific rate constant values obtained from Fig. 4 were fitted to Arrhenius equation (Eq. (5))

and the activation energy was estimated from the slope of the plot of $\ln k$ versus ($\frac{1}{T}$) as shown in Fig. 5.

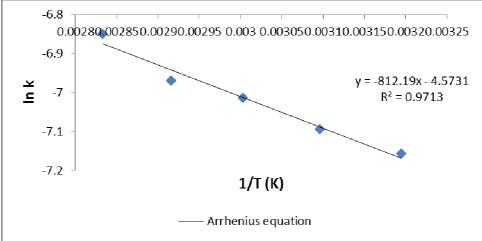


Fig. 5: Arrhenius equation fitted to the experimental data to estimate activation energy

The activation energy values suggests the ease with which hydrolysis can occur. A value of 6.75 kJ/mole was obtained for the banyan wood cellulose conversion to glucose. Activation energies of 26.6, 45, 76.71, 78.35, 80.34, 72.6, 64.35, 39.60, 37.83, 44.37 and 34.29 kJ/mole have correspondingly been obtained for the acid hydrolysis of sawdust, maize cobs, groundnut shells, sunflower seed hull, corn cob, corn fiber (a co-product of corn wet-milling), rice husk, banana skin, cowpea shells and maize stalks (Layokun, 1981; Aberuagba, 1997; Saracoglu et al., 1998; Mosier et al., 2002; Megawati et al., 2010; Ajani et al., 2012). The difference in the activation energies between these biomass celluloses can be attributed to difference in their degree of polymerization and crystallinity (i.e. structural features) (Aberuagba, 1997; Ajani et al., 2012). Crystallinity values have been reported to vary widely with a value of 73% in cotton linters and 29% in maize cobs (Ghose, 1956, Aberuagba, 1997). Furthermore, it has been reported that even when the ultimate chemical composition of two cellulosic materials is approximately the same, their response to cellulose attack can be surprisingly different (Ghose, 1956; Aberuagba, 1997; Ajani et al., 2012).

Conclusion

Acid hydrolysis of banyan wood cellulose was accomplished at different sulfuric acid concentrations (2 - 5 M), temperature (313 - 353 K) and reaction time (0 - 180 min). The acid hydrolysis of banyan wood cellulose increases with increase in acid concentration and temperature. The kinetics of concentrated acid hydrolysis of banyan wood cellulose can be quantitatively described by a pseudo first-order rate of homogenous reaction.

Kinetic constant can be expressed by Arrhenius equation with activation energy of 6.75 kJ/mole. Temperature and acid concentration strongly influences the rate of cellulose hydrolysis and thus the glucose yield. Therefore, from this work coupled with results of other researchers, it seems possible to totally convert agricultural biomass into useful products for the benefit of mankind.

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