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ACID HYDROLYSIS OF CORN COB TO GLUCOSE: A KINETIC STUDY

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Abstract: Corn cob is a renewable, cheap and abundant lignocellulosic material. Being a potential source of fermentable sugars, corn cob can be used as a substrate for value added products such as ethanol. This research work studied the glucose production from corn cob using phosphoric and nitric acids at 130°C, acid concentrations of 1wt%, 2wt%, 3wt%, 4wt% and 5wt% respectively and at time intervals of 18mins, 36mins, 54mins, 72mins and 90mins respectively. The substrate was characterized for the proximate constituents and the results show that corn cob has high hemicellulosic content. Seaman model and the Two-fraction model were used in studying the kinetics of glucose concentration in the hydrolysates, and the Two-fraction model gave a better fit with R^2 equal to 1. A maximum concentration of 0.038mg/ml of glucose was produced when 5wt% H_3PO_4 was used as catalyst for the hydrolysis, and a time interval of 42mins, with 0.4% susceptibility to hydrolysis. Whereas for the nitric acid hydrolysis, 0.054mg/ml maximum concentration of glucose was produced using 2wt% of HNO_3 , at a time interval of 77mins, with 0.6% susceptibility to hydrolysis. Comparatively, nitric acid proved to be a more efficient catalyst for acid hydrolysis of corn cobs.

Key Words: Corn cob, Acid hydrolysis, Glucose, Lignocellulosic material, Seaman's model, Two-fraction model.

I. INTRODUCTION

Agricultural residue can be referred to as lignocellulosic material. This is a low cost and the most abundant renewable energy source in the world. Examples of agricultural residues are corn cob, sugar cane bagasse, hardwood, softwood, corn stover, rice stover, wheat straw, and grasses etc. (Yuan et al., 2021).

With this economic advantage attached to lignocellulosic materials, it can be converted to useful products through hydrolysis (Wan et al., 2021). The hydrolysis of corn cobs to

produce glucose solutions could be a good alternative use for this abundant resource (Delbecqet al., 2018).

In diversifying the use of global renewable energy through better alternative sources there is need to convert agricultural residue which are in abundance to useful products such as ethanol (Guo et al., 2018). Acid hydrolysis being the method applied, this research work tends to determine between phosphoric and nitric acids which is preferable in the process of converting agricultural residual to useful products. Ethanol can serve as an alternative to transportation fuel, reduce greenhouse gas emission. There is need to properly understand the application of the kinetic models which were used to explain the variation with time of the main products generated and the optimal yield of glucose (Alessandra et al., 2012).

Corn cob is rich in glucose among all other lignocellulosic materials, making it a potential source of value-added products. Therefore, the hydrolysis of corn cob could be a good alternative for glucose production which could further be broken down to ethanol for further end use (Onyelucheya et al., 2016).

Economic interest in ethanol production can be enhanced if the required glucose solutions can be obtained from the hydrolysis of low-cost lignocellulosic wastes (Kalyaniet al., 2017).

This research paper focuses on the study of the concentration of glucose yield during acid hydrolysis of corn cob at a constant temperature of 130°C, for time intervals of 18mins, 36mins, 54mins, 72mins and 90mins and concentrations of 1wt%, 2wt%, 3wt%, 4wt% and 5wt % of both Phosphoric and Nitric acids (H_3PO_4 and HNO_3) respectively, and to determine the rate constants and other kinetic parameters of the individual acid hydrolysis process and to maximize production for the sugar of interest.

II. LITERATURE REVIEW AND THEORITICAL BACKGROUND

2.1 Kinetic Study of Lignocellulosic Material with Acids

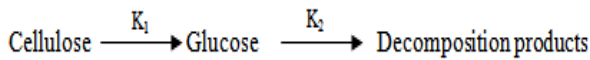
2.1.1 Kinetic Modeling of Lignocellulosic Material



Saeman and Two fraction models are the two kinetic models mostly proposed to investigate the acid hydrolysis of lignocellulosic materials by different researchers (Onyelucheyat et al., 2016).

2.1.2 Saeman's Model

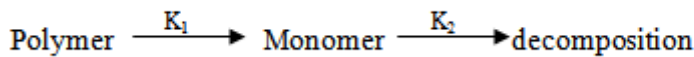
The simplified models for the study of the kinetics of hydrolysis process using acids began with the work of Saeman for the hydrolysis of douglas fire wood using sulphuric acid (Saeman, 1945). In his research the hydrolysis of cellulose was studied establishing the following model:



where $k_1(\text{min}^{-1})$ is the rate for release of glucose from cellulose and $k_2(\text{min}^{-1})$ is rate for glucose decomposition. This model considers the hydrolysis of cellulose to release glucose that in severe conditions is decomposed.

Eken-Saracglu et al. (1998) applied the Saeman's modes in their study done on kinetic comparative of hemicelluloses hydrolysis in corn cob and sun flower seed hull. The model was also applied in the works of Esther et al. (2012), for the hydrolysis of the hemicelluloses fraction of wheat straw using sulphuric acid. Onyelucheyat et al. (2016) in their study on acid hydrolysis of cassava peel applied similar models. Liang et al. (2017) in the effort to study the acid hydrolysis of corncobs to levulinic acid applied the Saeman's models. The models were helpful to da Costa Lopes&Lukasik (2018) in their work on separation and recovery of a hemicellulose-derived sugar produced from the hydrolysis of biomass by an acidic ionic liquid. Also, Wang et al. (2019) adopted the Saeman's models in their kinetic study on the hydrolysis of corncob residues to levulinic acid. And Yuan et al. (2021) equally applied the models in their kinetics studies on the hydrolysis of hemicellulose.

The model of Saeman can therefore be generalized for any polymer as:



The generalized polymer could be cellulose, hemicellulose, etc.

where K_1 = Rate of generation reaction and K_2 = Rate of decomposition reaction, and:

$$r_A = d \frac{C_A}{dt} = -k_1 C_A \tag{2.1}$$

$$r_B = d \frac{C_B}{dt} = -k_1 C_A - K_2 C_B \tag{2.2}$$

$$r_C = d \frac{C_C}{dt} = k_2 C_B \tag{2.3}$$

where C_A = Polymer concentration

C_B = Monomer concentration

C_C = Decomposition product concentration

From equation (2.1)

$$\begin{aligned} d \frac{C_A}{dt} &= -k_1 C_A \\ \int_{C_{A0}}^{C_A} \frac{dC_A}{C_A} &= -k_1 \int_0^t dt \\ \ln \frac{C_A}{C_{A0}} &= -K_1 t \end{aligned}$$

$$C_A = C_{A0} e^{-K_1 t} \tag{2.4}$$

Where; C_{A0} = Initial Polymer concentration

Substituting equation (2.1) into (2.2)

$$\frac{dC_B}{dt} = k_1 C_{A0} e^{-K_1 t} - k_2 C_B$$

$$\frac{dC_B}{dt} + k_2 C_B = k_1 C_{A0} e^{-K_1 t} \tag{2.5}$$

Equation (2.5) can be solved by Laplace transform or integrating factors.

Applying Laplace transform to equation (2.5);

$$\begin{aligned} L \left[\frac{dC_B}{dt} \right] + k_2 L[C_B] &= k_1 C_{A0} L[e^{-k_1 t}] \\ L \left[\frac{dC_B}{dt} \right] &= -k_2 L[C_B] + k_1 C_{A0} L[e^{-k_1 t}] \\ s C_B(s) - C_B(0) &= -k_2 C_B(s) + \frac{k_1 C_{A0}}{s + k_1} \end{aligned}$$

$C_B(0) = 0$ Because there is no monomer yet at time 0 sec.

$$s C_B(s) = -k_2 C_B(s) + \frac{k_1 C_{A0}}{s + k_1}$$

$$s C_B(s) + k_2 C_B(s) = \frac{k_1 C_{A0}}{s + k_1}$$

$$C_B(s) [s + k_2] = \frac{k_1 C_{A0}}{[s + k_1]}$$

$$C_B(s) = \frac{k_1 C_{A0}}{(s+k_1)(s+k_2)} \tag{2.6}$$

Resolve equation (2.6) into partial fraction

$$\frac{k_1 C_{A0}}{(s + k_1)(s + k_2)} = \frac{A}{(s + k_1)} + \frac{B}{(s + k_2)}$$

$$k_1 C_{A0} = A(s + k_2) + B(s + k_1)$$

Let $s = -k_1$

$$k_1 C_{A0} = A(k_2 - k_1)$$

$$A = \frac{k_1 C_{A0}}{(k_2 - k_1)} \tag{2.7}$$

Let $s = -k_2$

$$k_1 C_{A0} = A(-k_2 + k_2) + B(k_1 - k_2)$$

$$B = \frac{k_1 C_{A0}}{(k_1 - k_2)} \tag{2.8}$$

$$C_B(s) = \frac{k_1 C_{A0}}{(s+k_1)(s+k_2)} = \frac{k_1 C_{A0}}{(k_2-k_1)(s+k_1)} + \frac{k_1 C_{A0}}{(k_1-k_2)(s+k_2)} \tag{2.9}$$

$$C_B(s) = \frac{k_1 C_{A0}}{(k_2 - k_1)(s + k_1)} - \frac{k_1 C_{A0}}{(k_2 - k_1)(s + k_2)}$$



$$C_B(s) = \frac{k_1 C_{A0}}{(k_2 - k_1)} \left[\frac{1}{(s + k_1)} - \frac{1}{(s + k_2)} \right] \quad (2.10)$$

Find the Laplace inverse of equation (2.10)

$$C_B(t) = \frac{k_1 C_{A0}}{(k_2 - k_1)} [e^{-k_1 t} - e^{-k_2 t}] \quad (2.11)$$

where; t = time

C_{A0} can be determined analytically by equation (2.12)

$$C_{A0} = \frac{FZ\rho}{WSR} \quad (2.12)$$

Where;

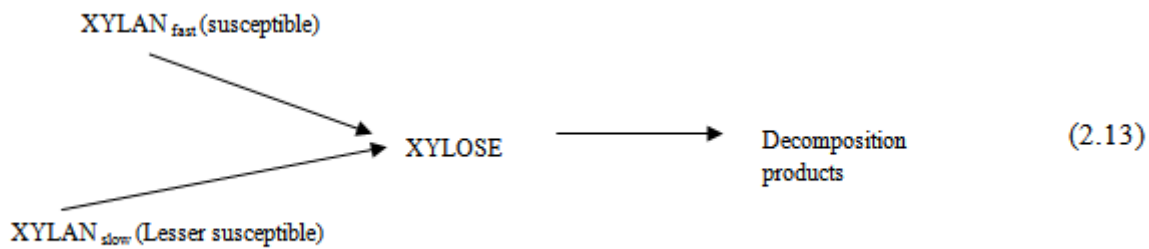
F= stoichiometric factor due to hydration of molecule during the hydrolysis

ρ = density of hydrolysate

z = composition of the raw material for the polysaccharides
WSR = water to solid ratio.

2.1.3 Two Fraction Model

Due to the limitation of Saeman's model to only one fraction of the polymer there was need to get an alternative called the Two fraction model which considered a susceptible or fast fraction and a less susceptible or slow fraction, as applied in the work of Aguilar et al. (2002) and da Costa Lopes & Lukasik (2018), where the Two fraction kinetic model showed a better fit as compared to the Saeman's model.



The ratio between the two fractions is the measured for the parameter α which is the mass fraction of the susceptible polymer in the raw material as shown in equation (2.14):

$$\alpha = \frac{\text{fast xylan}}{\text{total xylan}} \quad (2.14)$$

The lesser susceptible fraction does not react, remaining always in the solid phase. Then the equation that governs the kinetics is;

$$C_B(t) = \frac{\alpha k_1 C_{A0}}{(k_2 - k_1)} [e^{-k_1 t} - e^{-k_2 t}] \quad (2.15)$$

2.2 Comparing the Kinetic Parameters Based on Previous Research

2.2.1 Kinetic Modeling of Glucose Concentration

Glucose is the by-product obtained in the acid hydrolysis of lignocellulosic materials. Glucose released can either be from hemicellulose, heteropolymers and cellulose. Glucose

from cellulose is not usually hydrolysed in the range of operational condition for acid hydrolysis of hemicellulose (Onyelucheyat et al., 2016; Shi et al., 2017; Yuan et al., 2021).

A review of previous work that used similar substrate but different acids at constant temperature for glucose production is given below:

Aguilar et al. (2002) studied the kinetics for glucose concentration with sulfuric acid and using sugarcane bagasse as a substrate. They applied equation (2.12) to determine C_{A0} .

$$C_{A0} = \frac{Fz\rho}{WSR} \quad (2.12)$$

$$C_{A0} = \frac{180}{162} \times \frac{38.9}{10} \times 10 = 43.2 \text{ g/l}$$

They applied the two-fraction model to generate kinetic parameters, k_1, k_2 and α and also the statistical parameter (R^2) as shown in Table 2.1.

Table 2.1: Kinetic and statistical parameters of glucose released from the H_2SO_4 hydrolysis of sugarcane bagasse (Aguilar et al., 2002)

Operational set	α_g (g/g)	$k_1 \times 10^3$ (min ⁻¹)	$k_2 \times 10^3$ (min ⁻¹)	R^2
2% H ₂ SO ₄ at 122 °C	0.121	35.7	0.29	0.979
4% H ₂ SO ₄ at 122 °C	0.1460	84.2	0.42	0.997
6% H ₂ SO ₄ at 122 °C	0.182	74.1	0	0.995

k_1 = rate of generation reaction from glucan to glucose
 (min^{-1})
 k_2 = rate of decomposition reaction from glucose to HMF
 (min^{-1})

At 122°C, kinetic parameters of glucose generation tends to increase with increase in acid concentration whereas the decomposition reactions of glucose (k_2) are negligible. Sara et al. (2006) studied the hydrolysis of sugarcane bagasse using phosphoric acid. They applied the two-fraction model and generated kinetic parameters as shown in table 2.2.

Table 2.2 Results of fitting the kinetic models(Sara et al., 2006)

Operational set	α_G (g/g)	$k_1 \times 10^3$ (min^{-1})	$k_2 \times 10^3$ (min^{-1})	R^2
2% H_3PO_4 at 122 °C	0.09	2.38	0.000	0.99
4% H_3PO_4 at 122 °C	0.08	3.84	0.003	0.99
6% H_3PO_4 at 122 °C	0.08	4.90	0.305	0.99

From the table, their result followed similar trend with the work of Aguilar et al., (2002) which showed that the values of k_1 increased with the phosphoric acid concentration whereas the decomposition reactions of glucose (k_2) are negligible.

Antonio et al. (2004), in their work on kinetic assessment of sugarcane bagasse applied Saeman’s model and Two-fraction model, Two fraction models gave a better fit than the Seaman’s model as shown in Table 2.3.

Table 2.3: Results of the fitting for glucose concentration in the hydrolysis of sugar cane bagasse with nitric acid.(Antonio et al., 2004)

Operational set	Saeman’s model			Two-fraction model			
	$k_2 \times 10^3$	k_1 (min^{-1})	r^2	α_G (g/g)	k_1 (min^{-1})	$k_2 \times 10^3$ (min^{-1})	r^2
2% HNO_3 at 100°C	16.4	7.6	0.993	0.04	0.021	0.0	0.996
4% HNO_3 at 100°C	15.8	1.2	0.899	0.06	0.026	0.0	0.942
6% HNO_3 at 100°C	17.7	1.6	0.943	0.07	0.031	0.0	0.984
2% HNO_3 at 122°C	0.4	0.3	0.990	0.53	0.001	0.0	0.990
4% HNO_3 at 122°C	3.6	1.4	0.859	0.16	0.011	0.0	0.871
6% HNO_3 at 122°C	22.9	3.8	0.801	0.10	0.127	0.3	0.997
2% HNO_3 at 128°C	12.7	0.9	0.811	0.21	0.004	6.3	0.888
4% HNO_3 at 128°C	14.9	2.0	0.761	0.09	0.042	0.1	0.999
6% HNO_3 at 128°C	12.6	2.0	0.780	0.10	0.034	0.0	0.976

The first three sets are at operating conditions of temperature of 100°C and acid concentration at 2wt%, 4wt% and 6wt% HNO_3 respectively. The next three sets are at operating conditions of 122°C and acid concentration at 2wt%, 4wt% and 6wt% HNO_3 respectively. The last three sets are at operating condition of 128°C and at 2wt, 4wt and 6wt% HNO_3 respectively. They had similar conclusion with that of Aguilar et al. (2002), they concluded that k_1 increased with nitric acid concentration but the decomposition of glucose was negligible, being the coefficient affected either by temperature or nitric acid concentration.

effectively reduce the moisture content and surface wetness of the raw sample.



Figure 3.1: Fresh corn cobs undergoing sun drying

III. MATERIALS AND METHOD

3.1 Material acquisition

The corn cobs used for the experiments were gathered from local farmers in Obodoukwu Community in Ideya to North LGA of Imo State, Nigeria. The fresh corn cobs (as shown in Fig 3.1) were gathered, sun dried for about three days, to

3.2 Pretreatment of Fresh Corn Cobs

After the fresh corn cobs has been sun-dried, they were taken to the grinding machine for particle size reduction to

powdered form. The ground corn cobs (hydrolysis substrate) were then taken to laboratory, screened through a 1.0 mm mesh size, as shown in Fig 3.2, and stored in a container in a cool dry place, from where samples were taken and analyzed to determine some proximate parameters cellulose, hemicellulose, lignin, ash, carbohydrate and the moisture content.



Figure 3.2: Screened Corn Cobs

3.3 Acid Hydrolysis of the Corn Cobs

20g of the sample (substrate) was weighed into a 500ml conical flask (reaction vessel). 400ml of phosphoric acid solution (H_3PO_4) was then added and the mixture swirled carefully, placed on an electro-thermal magnetic stirrer, set at 150rpm and at a temperature of $130^{\circ}C$ and at the pre chosen time. At the end of the reaction time, the reaction mixture was quenched in a very cold ice-bath. After 2minutes, the content of the conical flask was filtered and labeled appropriately (with regards to reaction time conditions) and kept in a safe place for the glucose tests. The filtrate samples were afterwards taken to the uv-spectrophotometer for the absorbance readings, at a wavelength of 540nm. For the filtrate sample preparations, DNS reagent was used as outlined in the standard analytical procedures

The same procedure was used for the nitric acid (HNO_3) hydrolysis and reducing sugar test preparations, at different operating time conditions of 18, 36, 54, 72 and 90minutes respectively at a temperatures of $130^{\circ}C$, using five different acid concentrations of 1% w/w, 2% w/w, 3% w/w, 4% w/w and 5% w/w respectively.

3.4 Calibration of Absorbance Reading

0.2g of glucose was weighed and 100ml of distilled water was added to it to get a stock solution concentration of 20mg/dl. 0.1ml, 0.2ml, 0.3ml, 0.4ml, 0.5ml and 0.6ml were taken with the aid of pipette into six test tubes. A test tube containing blank solution was also prepared. 0.9ml, 0.8ml, 0.7ml, 0.6ml, 0.5ml, and 0.4ml of distilled water were added to each test tube. Also 3ml of DNS reagent was added to each test tube and it was covered with aluminum foil, and the test tubes taken to the hot water bath and heated for 5minutes. Afterwards, 1ml of sodium potassium tartrate was

added to each test tube and taken to spectrophotometer set at 540nm wavelength to determine the absorbance reading of glucose at the different concentrations.

All experiments were carried out in duplicates and their average values were used to obtain the concentration values, from the glucose standard calibration curves.

3.5 Procedure for Absorbance Readings

The spectrophotometer was switched on and allowed to boot for 30mins before use. The equipment was set to the measuring wavelength (for the sample) of the transmitting light and at 100% transmittance. The cuvettes were cleaned and filled to 4ml of the blank solution, and was inserted into the equipment to calibrate the machine at the set wavelength for that sample, with the blank solution. The blank solution was removed and in turn the cuvette was filled with the sample solutions and insert into the machine to obtain the absorbance readings for the sample solutions, at the set wavelength.

3.6 Procedure for Obtaining the Kinetic and Statistical Parameters

The experimental results were converted from absorbance (nm) to concentrations with the aid of the calibration data given.

The concentration values were plotted against time (in minutes). The experimental data were fit to both kinetic models (Saeman & Two fraction) for phosphoric acid and nitric acid with a curve fitting tool in MatLab software, to obtain kinetic parameters (k_1 and k_2), α and statistical parameter (*Adjusted R²*). where k_1 is the rate of monomer generation reaction, k_2 is the rate of monomer decomposition reaction while α is the mass ratio of susceptible polymer fraction to total polymer (kg kg₋₁).

IV. RESULTS AND DISCUSSION

4.1 Results presentation

The composition of the corn cob used in this study is shown in Table 4.1. The main fractions of corn cob were in the same range as other herbaceous materials, such as rice, wheat straw and sorghum straw.

To obtain relationship between absorbance and concentration with the aid of curve fitting tool in excel, the absorbance (nm) values were plotted against concentration (mg/dl) values shown in Table 4.2 to obtain the standard calibration curve.



Components	Mass fraction (%)
Hemicellulose	42.36
Cellulose	31.26
Lignin	12.25
Ash	1.85
Carbohydrate	2.86
Moisture content	3.35
Others	6.07

Table 4.1: Main component of corn cob used in this study

Time (min)	G ₁ (nm)	G ₂ (n m)	G _{average} (nm)	G (mg/ml)
0	0	0	0	0
18	0.914	0.910	0.912	0.0325
36	1.156	0.986	1.071	0.0382
54	0.979	0.983	0.981	0.0351
72	0.840	0.862	0.851	0.0304
90	0.912	0.838	0.875	0.0312

Table 4.4: Experimental result for the glucosereleased at 2wt% phosphoric acid

Absorbance (nm)	Concentration (mg/dl)
0	0.0000
0.2	0.1460
0.4	0.3255
0.6	0.4060
0.8	0.4505
1.0	0.4695

Table 4.2: Calibration data for glucose (540nm)

Time (min)	G ₁ (nm)	G ₂ (n m)	G _{average} (nm)	G (mg/ml)
0	0	0	0	0
18	0.842	0.864	0.853	0.0305
36	1.125	0.917	1.021	0.0364
54	0.995	1.025	1.010	0.0361
72	0.936	0.904	0.920	0.0329
90	0.805	0.813	0.809	0.0289

Table 4.5: Experimental result for the glucose released at 3wt% phosphoric acid

4.1.1 Experimental Result for Glucose Using Phosphoric Acid

The experimental results of glucose concentration after conversion of its absorbance reading for phosphoric acids are given in Tables 4.3, 4.4, 4.5, 4.6, and 4.7.

Time (min)	G ₁ (nm)	G ₂ (n m)	G _{average} (nm)	G (mg/ml)
0	0	0	0	0
18	0.873	0.865	0.873	0.0312
36	1.036	1.038	1.037	0.0371
54	0.978	0.965	0.969	0.0346
72	0.839	0.843	0.841	0.0305
90	0.709	0.693	0.701	0.0254

Table 4.6: Experimental result for the glucose released at 4wt% phosphoric acid

Time (min)	G ₁ (nm)	G ₂ (n m)	G _{average} (nm)	G (mg/ml)
0	0	0	0	0
18	0.926	0.908	0.917	0.0327
36	1.023	1.087	1.055	0.0376
54	0.945	1.049	0.997	0.0356
72	0.812	0.818	0.815	0.0291
90	0.777	0.773	0.775	0.0277

Table 4.3: Experimental result for the glucose released at 1wt% phosphoric acid

Time (min)	G ₁ (nm)	G ₂ (n m)	G _{average} (nm)	G (mg/ml)
0	0	0	0	0
18	0.859	0.865	0.862	0.0308
36	1.040	1.042	1.041	0.0371
54	1.014	1.010	1.012	0.0362
72	0.957	0.965	0.961	0.0344
90	0.803	0.789	0.796	0.0285

Table 4.7: Experimental result for the glucose released at 5wt% phosphoric acid



Where;

G_1 = First absorbance reading

G_2 = Second absorbance reading

G = Concentration of glucose in mg/ml

The Kinetic and statistical parameter of glucose released, applying both Seaman's model and Two-fraction model using phosphoric acid are shown in tables 4.8 & 4.9 below.

Conc (wt%)	K_1 (min^{-1})	K_2 (min^{-1})	Adj R^2
1	0.003451	1.257	0.8329
2	0.001575	0.6082	0.9552
3	0.0003283	0.1346	0.7540
4	0.003137	1.177	0.9312
5	0.0003196	0.1292	0.8441

Table 4.8: Kinetic and statistical parameter of glucose released for Seaman's model using phosphoric acid

Conc (wt%)	K_1 (min^{-1})	K_2 (min^{-1})	α	Adj R^2
1	0.9035	0.003451	0.02741	0.9105
2	0.7644	0.002803	0.02684	0.925
3	0.08389	0.005148	0.03169	0.982
4	0.05453	0.01124	0.04002	0.9758
5	0.05057	0.008932	0.03917	0.9964

Table 4.9: Kinetic and statistical parameter of glucose released for Two fraction model using phosphoric acid

To compare the experimental values with values obtained applying the Saeman's model and Two-fraction model at varying concentrations of phosphoric acid for glucose released is shown below.

Time (min)	Experimental Data (mg/ml)	Saeman's Model (mg/ml)	Two-fraction model (mg/ml)
0	0	0	0
18	0.0327	0.02608	0.03606
36	0.0376	0.03390	0.03789
54	0.0356	0.03186	0.03484
72	0.0291	0.02654	0.02993
90	0.0277	0.02614	0.02812

Table 4.10: Comparison of the experimental data with the model at 1wt% phosphoric acid for the glucose released

Time (min)	Experimental Data (mg/ml)	Saeman's Model (mg/ml)	Two-fraction model (mg/ml)
0	0	0	0
18	0.0325	0.03119	0.03572
36	0.0382	0.02921	0.03896
54	0.0351	0.03225	0.03429
72	0.0304	0.02232	0.03069
90	0.0312	0.02142	0.02919

Table 4.11: Comparison of the experimental data with the model at 2wt% phosphoric acid for the glucose released

Time (min)	Experimental Data (mg/ml)	Saeman's Model (mg/ml)	Two-fraction model (mg/ml)
0	0	0	0
18	0.0305	0.02487	0.03051
36	0.0364	0.03342	0.03682
54	0.0361	0.03347	0.03514
72	0.0329	0.02629	0.03268
90	0.0289	0.0231	0.02959

Table 4.12: Comparison of the experimental data with the model at 3wt% phosphoric acid for the glucose released

Time (min)	Experimental Data (mg/ml)	Saeman's Model (mg/ml)	Two-fraction model (mg/ml)
0	0	0	0
18	0.0312	0.03522	0.03108
36	0.0371	0.03328	0.03703
54	0.0346	0.03146	0.03461
72	0.0305	0.02973	0.02991
90	0.0254	0.02809	0.02504

Table 4.13: Comparison of the experimental data with the model at 4wt% phosphoric acid for the glucose released



Time (min)	Experimental Data (mg/ml)	Saeman's Model (mg/ml)	Two-fraction model (mg/ml)
0	0	0	0
18	0.0308	0.031	0.02979
36	0.0371	0.03385	0.03735
54	0.0362	0.03395	0.03663
72	0.0344	0.03379	0.03313
90	0.0285	0.03359	0.02899

Table 4.14: Comparison of the experimental data with the model at 5wt% phosphoric acid for the glucose released

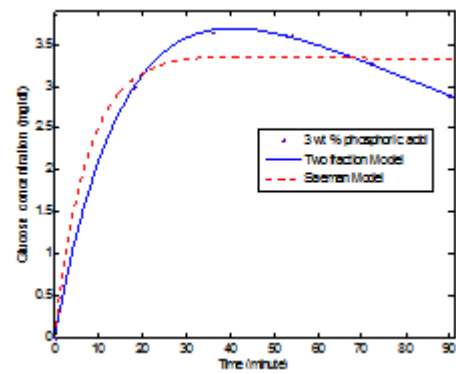


Figure 4.3: The graph for glucose released at 3wt% phosphoric acid

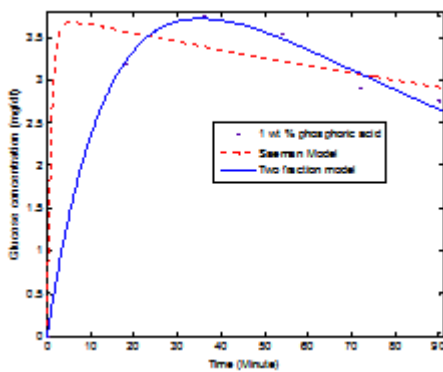


Figure 4.1: The graph for glucose released at 1wt% phosphoric acid

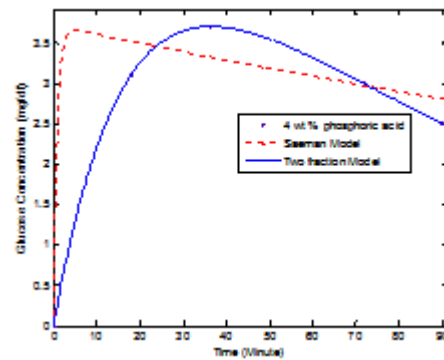


Figure 4.4: The graph for glucose released at 4wt% phosphoric acid

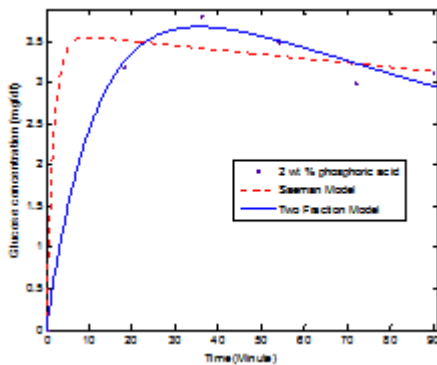


Figure 4.2: The graph for glucose released at 2wt% phosphoric acid

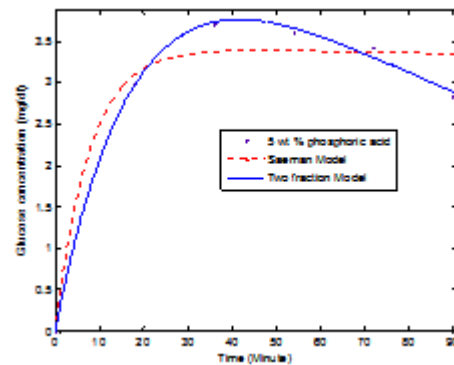


Figure 4.5: The graph for glucose released at 5wt% phosphoric acid

4.1.2 Experimental Result for Glucose Using Nitric Acid

The experimental results of glucose concentration after conversion of its absorbance reading for nitric acids are given in Tables 4.15, 4.16, 4.17, 4.18, and 4.19.



Time (min)	G ₁ (nm)	G ₂ (n m)	G _{average} (nm)	G (mg/ml)
0	0	0	0	0
18	0.760	0.744	0.752	0.0268
36	1.216	1.015	1.116	0.0398
54	1.541	1.532	1.535	0.0548
72	1.546	1.546	1.546	0.0552
90	1.408	1.404	1.406	0.0502

Table 4.15: Experimental result for the glucose released at 1wt% nitric acid

Time (min)	G ₁ (nm)	G ₂ (n m)	G _{average} (nm)	G (mg/ml)
0	0	0	0	0
18	0.932	0.712	0.822	0.0293
36	1.343	1.343	1.343	0.0479
54	1.345	1.351	1.348	0.0481
72	1.350	1.351	1.3505	0.0482
90	1.352	1.353	1.3525	0.0483

Table 4.19: Experimental result for the glucose released at 5wt% nitric acid

Time (min)	G ₁ (nm)	G ₂ (n m)	G _{average} (nm)	G (mg/ml)
0	0	0	0	0
18	0.562	0.556	0.559	0.0199
36	1.295	1.275	1.285	0.0459
54	1.542	1.542	1.542	0.055
72	1.482	1.490	1.486	0.053
90	1.422	1.434	1.428	0.0509

Table 4.16: Experimental result for the glucose released at 2wt% nitric acid

Time (min)	G ₁ (nm)	G ₂ (n m)	G _{average} (nm)	G (mg/ml)
0	0	0	0	0
18	0.363	0.355	0.359	0.0128
36	0.427	0.423	0.425	0.0152
54	0.420	0.425	0.4225	0.0151
72	0.424	0.426	0.425	0.0152
90	0.424	0.426	0.425	0.0152

Table 4.17: Experimental result for the glucose released at 3wt% nitric acid

Time (min)	G ₁ (nm)	G ₂ (n m)	G _{average} (nm)	G (mg/ml)
0	0	0	0	0
18	0.395	0.395	0.395	0.0141
36	0.426	0.425	0.426	0.0352
54	0.422	0.428	0.425	0.0452
72	1.349	1.352	1.351	0.0482
90	1.347	1.350	1.349	0.0441

Table 4.18: Experimental result for the glucose released at 4wt% nitric acid

Where;

G₁ = First absorbance reading

G₂ = Second absorbance reading

G = Concentration of glucose in mg/ml

The Kinetic and statistical parameter of glucose released, applying both Seaman's model and Two-fraction model using nitric acid are shown in tables 4.20 & 4.21 below.

Conc (wt%)	K ₁ (min ⁻¹)	K ₂ (min ⁻¹)	Adj R ²
1	9.446e-005	0.03808	0.866
2	8.955e-005	0.03534	0.8386
3	6.878e-005	0.1031	0.7993
4	6.545e-005	0.02772	0.8176
5	0.0001252	0.05708	0.9813

Table 4.20: Kinetic and statistical parameter of glucose released for Seaman's model using nitric acid

Conc (wt%)	K ₁ (min ⁻¹)	K ₂ (min ⁻¹)	α	Adj R ²
1	0.01326	0.01325	0.06351	0.9744
2	0.01301	0.01302	0.06379	0.9455
3	0.09755	0.0004257	0.06817	0.9993
4	0.01112	0.01114	0.0569	0.9126
5	0.03497	0.005508	0.03072	0.985

Table 4.21: Kinetic and statistical parameter of glucose released for Two fraction model using nitric acid

To compare the experimental values with values obtained applying the Saeman's model and Two-fraction model at varying concentrations of nitric acid for glucose released is show below.



Time (min)	Experimental Data (mg/ml)	Saeman's Model (mg/ml)	Two-fraction model (mg/ml)
0	0	0	0
18	0.0268	0.02818	0.02736
36	0.0398	0.04234	0.03911
54	0.0548	0.04941	0.05394
72	0.0552	0.05293	0.05551
90	0.0502	0.05465	0.05269

Table 4.22: Comparison of the experimental data with the model at 1wt% nitric acid for the glucose released

Time (min)	Experimental Data (mg/ml)	Saeman's Model (mg/ml)	Two-fraction model (mg/ml)
0	0	0	0
18	0.0141	0.02124	0.02136
36	0.0352	0.03412	0.03497
54	0.0452	0.04191	0.04293
72	0.0482	0.04661	0.04785
90	0.0441	0.04944	0.04393

Table 4.25: Comparison of the experimental data with the model at 4wt% nitric acid for the glucose released

Time (min)	Experimental Data (mg/ml)	Saeman's Model (mg/ml)	Two-fraction model (mg/ml)
0	0	0	0
18	0.0199	0.0273	0.02038
36	0.0459	0.04172	0.04285
54	0.055	0.0493	0.05086
72	0.053	0.05327	0.05365
90	0.0509	0.05533	0.05305

Table 4.23: Comparison of the experimental data with the model at 2wt% nitric acid for the glucose released

Time (min)	Experimental Data (mg/ml)	Saeman's Model (mg/ml)	Two-fraction model (mg/ml)
0	0	0	0
18	0.0293	0.02223	0.03015
36	0.0479	0.04369	0.0468
54	0.0481	0.04773	0.04942
72	0.0482	0.04509	0.04947
90	0.0483	0.04352	0.04731

Table 4.26: Comparison of the experimental data with the model at 5wt% nitric acid for the glucose released

Time (min)	Experimental Data (mg/ml)	Saeman's Model (mg/ml)	Two-fraction model (mg/ml)
0	0	0	0
18	0.0128	0.01289	0.01286
36	0.0152	0.01429	0.01498
54	0.0151	0.01518	0.01525
72	0.0152	0.01521	0.01520
90	0.0152	0.01520	0.01509

Table 4.24: Comparison of the experimental data with the model at 3wt% nitric acid for the glucose released

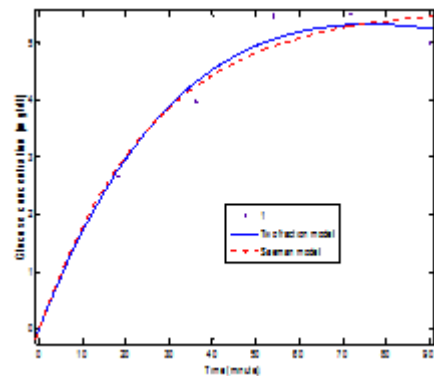


Figure 4.6: The graph for glucose released at 1wt% nitric acid

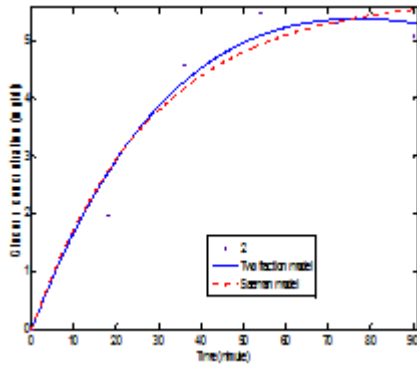


Figure 4.7: The graph for glucose released at 2wt% nitric acid

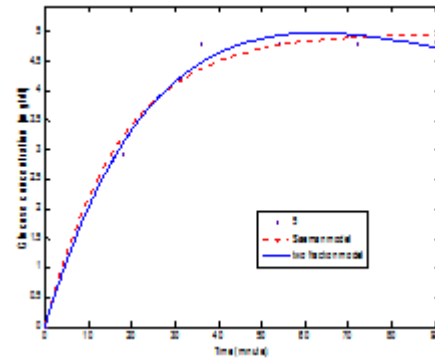


Figure 4.10: The graph for glucose released at 5wt% nitric acid

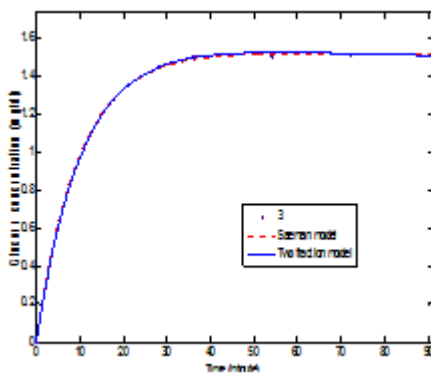


Figure 4.8: The graph for glucose released at 3wt% nitric acid

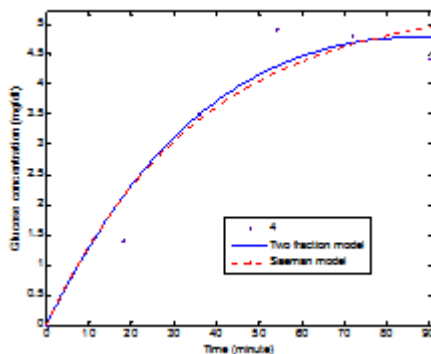


Figure 4.9: The graph for glucose released at 4wt% nitric acid

4.2 Discussion of Results

Table 4.1 show the result of proximate analysis of the corn cob used in this study, this analysis shows that it is richer in hemicellulose than cellulose.

The experimental result of glucose concentration for phosphoric acid are given in Tables 4.3, 4.4, 4.5, 4.6, 4.7 and that for nitric acid are given in Tables 4.15, 4.16, 4.17, 4.18, and 4.19 respectively. They were converted from absorbance (nm) to concentration (mg/dl) with the aid of calibration data given in Table 4.2. Also the experimental data were fit to Saeman's model and Two-fraction model.

The data of Table 4.2 was used to obtain the standard calibration curve used for the conversion of the absorbance data (nm) to concentration data (mg/ml), with the help of equation 4.1 showing the relationship between the two variables.

$$\text{Concentration} = \left[\frac{\text{Absorbance}}{0.5604} \right] \times 2 \quad (4.1)$$

Equation (4.1) was used to convert absorbance reading in column four of Table 4.3, 4.4, 4.5, 4.6 and 4.7 for phosphoric acid and Tables 4.15, 4.16, 4.17, 4.18, and 4.19 for nitric acid respectively to concentration in column five of the same tables.

Equation 4.2 is the general Saeman's model equation whereas equations 4.3 and 4.4 are Saeman model used for phosphoric and nitric acids hydrolysis respectively, to yield glucose.

$$C_B(t) = \frac{CA_0K_1}{(k_2 - k_1)} [e^{-k_1t} - e^{-k_2t}] \quad (4.2)$$

$$G(t) = \frac{10.5669K_1}{(K_2 - k_1)} [e^{-k_1t} - e^{-k_2t}] \quad (\text{H}_3\text{PO}_4) \quad (4.3)$$

$$G(t) = \frac{14.0644K_1}{(k_2 - k_1)} [e^{-k_1t} - e^{-k_2t}] \quad (\text{HNO}_3) \quad (4.4)$$

Glucose concentrations (mg/ml) were plotted from Table 4.3, 4.4, 4.5, 4.6 and 4.7 for phosphoric acid and Tables 4.15, 4.16, 4.17, 4.18 and 4.19 for nitric acid respectively against time (min), the experimental data were fit to



equation (4.3) for phosphoric acid and equation (4.4) for nitric acid with curve fitting tool in MatLab software to obtain kinetic parameters (k_1 and k_2) and statistical parameter (*Adjusted R*²) as shown in Tables 4.8 and 4.20. The figures for the plots are shown in Figures 4.1, 4.2, 4.3, 4.4, 4.5, 4.6, 4.7, 4.8, 4.9 and 4.10, for both phosphoric and nitric acids respectively. The values of k_1 and k_2 were substituted into equation 4.3 and 4.4 to generate Seaman's model data at different concentrations of phosphoric and nitric acids, given in column three of Tables 4.10, 4.11, 4.12, 4.13 and 4.14 for phosphoric acid and Tables 4.22, 4.23, 4.24, 4.25 and 4.26 for nitric acid, respectively. Equation 4.4 is the general Two-fraction model equation whereas equations 4.6 and 4.7 are Two-fraction model used for phosphoric and nitric acids hydrolysis respectively, to yield glucose.

$$C_B(t) = \frac{CA_0 aK_1}{(k_2 - k_1)} [e^{-k_1 t} - e^{-k_2 t}] \quad (4.5)$$

$$G(t) = \frac{10.5669 aK_1}{(k_2 - k_1)} [e^{-k_1 t} - e^{-k_2 t}] \quad (H_3PO_4) \quad (4.6)$$

$$G(t) = \frac{14.0644 aK_1}{(k_2 - k_1)} [e^{-k_1 t} - e^{-k_2 t}] \quad (HNO_3) \quad (4.7)$$

Glucose concentrations (mg/ml) were plotted from Table 4.3, 4.4, 4.5, 4.6 and 4.7 for phosphoric acid and Tables 4.15, 4.16, 4.17, 4.18, and 4.19 for nitric acid respectively against time (min), the experimental data were fit to equation (4.6) for phosphoric acid and equation (4.7) for nitric acid with curve fitting tool in MatLab to obtain kinetic parameters (k_1 and k_2), α and statistical parameter (*Adjusted R*²), as shown in Table 4.9 and 4.21. The figures for the plots are shown in Figures 4.1, 4.2, 4.3, 4.4, 4.5, 4.6, 4.7, 4.8, 4.9 and 4.10, for both phosphoric and nitric acids respectively. The values of k_1 , k_2 and α were substituted into equation 4.6 and 4.7 to generate Two-fraction model data at different concentrations of phosphoric acid and nitric acids, given in column four of Tables 4.10, 4.11, 4.12, 4.13 and 4.14 for phosphoric acid and Tables 4.22, 4.23, 4.24, 4.25 and 4.26 for nitric acid, respectively.

The glucose concentrations were also fitted into both the Saeman and Two-fraction models for the phosphoric acid hydrolysis as shown in Tables 4.8 and 4.9. Comparing the values of the statistical parameter (*Adjusted R*²), it was found that the Two-fraction model fits better. This result agrees with those of Aguilar et al. (2002), Onyeluchey et al. (2016), Wang et al. (2019) and Yuan et al. (2021).

It is noteworthy that from Table 4.9, the values of K_1 were higher than those of K_2 simply showing that the rate of glucose generation is high compared to its decomposition which is slow. This is in line with the findings of the work of Esther et al. (2012), Shi et al. (2017) and Santos et al. (2018) and Yuan et al. (2021).

The value of α is in the range of 0.02-0.04 mg/mg with an average of 0.03 mg/mg indicating that 0.3% glucan was susceptible to hydrolysis which is smaller as compared to the works of Aguilar et al. (2002) and da Costa Lopes & Lukasik (2018). This was also affected by acid concentration as it increased with an increase in the concentration of the acid.

The glucose concentrations were again fitted into both the Saeman and Two-fraction models for the nitric acid hydrolysis as shown in Tables 4.20 and 4.21. Comparing the values of the statistical parameter (*Adjusted R*²), and it was again found that the two-fraction model fits better. This result also agrees with those of Aguilar et al. (2002), Onyeluchey et al. (2016) and Yuan et al. (2021).

The kinetic parameters of decomposition reactions of glucose K_2 is in the same range of K_1 . It can be supposed that this is due to high rate of degradation of glucose. This is in line with the findings in the work of Aguilar et al. (2002) and Negahdaret al. (2016).

The parameter α was in the range of 0.03-0.06 mg/mg. it was slightly affected by increase in acid concentration, as also suggested by Onyeluchey et al. (2016) and Wang et al. (2019).

To substantiate the fact that the Two-fraction model is better compared to the Saeman's model, from Tables 4.10, 4.11, 4.12, 4.13, 4.14, 4.22, 4.23, 4.24, 4.25 and 4.26, comparing their third column (Saeman's model) with the fourth column (Two-fraction model) of the same Tables, the values from the fourth column are almost always closer to the experimental values.

The maximum glucose production was predicted as 5wt% H_3PO_4 for 42mins, which gave 0.038mg/ml with 0.4% susceptibility to hydrolysis. Whereas the maximum glucose production was predicted as 2wt% HNO_3 for 77mins, which gave 0.054mg/ml with 0.6% susceptibility to hydrolysis.

Comparing these results with the maximum production obtained by Laboratory confirmation test; for glucose production at 5wt% H_3PO_4 for 42mins, gave 0.037mg/ml, where as using nitric acid as a catalyst, glucose production at 2wt% HNO_3 for 77mins, gave 0.046mg/ml of glucose.

And comparing these results with the maximum production obtained by Laboratory confirmation test and previous researchers; Antonio et al. (2004) in their "hydrolysis of sugarcane bagasse using nitric acid", the maximum conditions selected were 122°C, 6wt% and 9.3mins which gave 2.87mg/ml of glucose. Yuan et al. (2021) confirmed these conditions with similar result in their recent research.

Sara et al. (2006) in their work, "Study of the hydrolysis of sugar cane bagasse using phosphoric acid", had maximum conditions selected at 122°C, 4wt% and 300mins, which gave 3.0mg/ml of glucose, which has been corroborated by the findings of Shi et al. (2017).

Esther et al. (2012), in their study on "Acid hydrolysis of wheat straw" using sulphuric acid as a catalyst, the maximum conditions selected were 2wt% of H_2SO_4 at



130°C for 29mins, which gave glucose concentration of 3.5mg/ml with 11% susceptibility. And On yelucheya et al. (2016) in their acid hydrolysis of cassava peel using phosphoric acid, the maximum glucose concentration was obtained as 2.218mg/ml at 7.5wt%, 1min and 121°C.

From the results compared above, the glucose concentration is low, indicating a small degradation of the cellulosic fraction.

V. CONCLUSION

Kinetic study of the acid hydrolysis of corn cob to determine the yield of glucose has been established in this work. It has shown that corn cob is richer in hemicellulose content than cellulose, and this made it a weak source of glucose. Again, it has been shown that the rate of decomposition of the polymer (K_1) is greater than the rate of degradation of the monomer (K_2). Two-fraction model is found to be a better model for fitting of the hydrolysis data, as it gave better fits over the Seaman's model, and the parameter α is less than or equal to 1 and is slightly affected by increase in acid concentration.

A maximum concentration of 0.038 mg/ml glucose was produced when 5wt% H_3PO_4 was used as catalyst for the hydrolysis, and a time interval of 42mins, with 0.4% susceptibility to hydrolysis, whereas for the nitric acid hydrolysis, 0.054mg/ml maximum glucose concentration was produced using 2wt% of the nitric acid, at a time interval of 77mins, with 0.6% susceptibility to hydrolysis. And comparing the results obtained using phosphoric acid and nitric acid, it has been established that the nitric acid is a more efficient catalyst for the hydrolysis.

Also, the results obtained from this research within the already established operational conditions shows that corn cob as a lignocellulosic material is suitable for the production of glucose.

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