# The Kinetics of Hydrolysis of Breadfruit Husks using HCl

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**Abstract**

The aim of this work was to study the effects of process parameters on the kinetics of the acidic hydrolysis of breadfruit husks. Breadfruit husks were obtained from dumpsite close to Oye Market in Emene, Enugu State Nigeria. The husks were pretreated and characterized, and it was found to contain 46% hydrolysable celluloses and hemicelluloses. In view of the appreciable amount of hydrolysable components, and thus its potential use for bioethanol production, the kinetics study, a path way to process design and sizing of equipment, was deemed necessary. The kinetics of the hydrochloric acid hydrolysis was influenced by temperature and acid concentration, hence, One-Factor-at- a- Time (OFAT) method of experiment was applied to study the effect of the process parameters. The kinetics was studied at 70, 90, 150 and 190 °C with 0.1M, 0.5M and 1.0M acid concentrations. At low temperature, the maximum conversion varied linearly with the acid concentration but as the temperature increased above 90 °C, the maximum conversion started decreasing with increase in acid concentration. Temperature equally had a direct proportional relationship with the conversion, but the trend reversed above 150°C. The highest conversion of 67% was recorded at 150°C, 0.5M and 3hrs. The kinetics parameters showed that the rate of sugar formation was higher than the rate of sugar decomposition at temperature above 150°C and 90°C for acid concentrations of 0.1M and 0.5–1.0M respectively. The thermodynamic parameters indicated positive values of the enthalpy change, which confirms that heat was needed for both sugar formation and decomposition processes. The entropy changes were negative showing that both the sugars and the decomposition products were stable. The result also showed that sugars formed with low acid concentration were more stable than those formed with high acid concentrations.

**Index Terms-** Breadfruit husks, Hydrolysis, Kinetics, Sugar, Thermodynamics

## 1 INTRODUCTION

Governments and private institutions in different parts of the world are currently exploring technologies and means of creating alternative energy sources to meet the energy demands of increasing human population [1]. Due to the depletion of fossil fuel resources, environmental pollution concerns, the high cost of conventional energy sources, and the need to diversify energy resources to meet the increasing population and industrialization, bio and renewable energy sources are of global interest. The escalating demand for alternative energy sources attracted the use of biofuel, which can be bioethanol, biogas, or biodiesel. Biofuels can be classified as first generation or second generation; first generation biofuels are derived from carbohydrates, lipids, and oil while the second generation is derived from lignocellulosic biomass such as crop peels, husks, shells, wood etc. [2]. The conventional crops or the first generation biofuel sources are unable to meet the global demand due to their primary values of food and feed [3]. Consequently, in developing countries, food-related feedstock is preferably replaced by lignocelluloses[4].

Bioethanol, a liquid, and colorless biofuel have been described as an attractive biofuel having the potential for energy security and environmental safety over fossil fuel [5]. In Nigerian context, moreover, the key drivers to bioethanol demand are the urgent need to reduce energy insecurity, need to increase electricity accessibility, need to raise Gross Domestic Product (DGP), to maximize the use of available resources and the urgent need to reduce unemployment [6]. Currently, the Nigerian Government is in partnership with Brazil for bioethanol production using cassava, sweet sorghum and sugarcane as feedstock with a set target of 1.27 billion liters per year. This development would at long run cause more harm to the food security of this nation where it is already estimated that over 300 persons die every year as a result of hunger [7].

Lignocellulose, a by-product of agricultural activities that are entirely outside the food chain of humans could offer a better alternative. Lignocellulose is the principal component of the plant cell walls and is mainly composed of cellulose (40–60% of the total dry weight), hemicellulose (20–40%), and lignin (10–25%) [5]. Lignocellulosic biomass can be pretreated and hydrolysed to release sugars which can be fermented to bioethanol in a downstream process similar to that of first generation feedstock [2]. There are several possible methods to hydrolyze lignocelluloses. The most commonly applied methods can be classified into two groups: chemical hydrolysis and enzymatic hydrolysis. However, there are some other hydrolysis methods in which no chemicals or enzymes are applied. For instance, lignocelluloses may be hydrolyzed by gamma-ray or electron-beam irradiation, or microwave irradiation but these processes are far from being commercially applied [8]. The Enzymatic hydrolysis usually gives a high yield of simple sugars at mild operating conditions but the rate of hydrolysis is always lower than that of the chemical hydrolysis and the high cost is also a challenge [9]. However, many agricultural wastes lignocelluloses such as rice straw, sugarcane bagasse, corncobs, corn stover, wheat straw have been recorded as potential feed stock for ethanol production through enzymatic hydrolysis [10] [11]. Among the chemical hydrolysis method, concentrated and dilute acid methods are the most commonly studied and applied. The concentrated acid method, though reportedly yields a high concentration of simple sugars, the high maintenance and investment costs have greatly reduced the potential commercial interest of the process [8]. The dilute

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acid hydrolysis process is usually employed at a concentration of 1-10% at temperatures in the range of 100-150°C [2]. Quite reasonable research findings have been recorded on the conversion of agricultural wastes into simple sugars via dilute acid hydrolysis using pressured vessels. Lenihan et al. [9] recorded sugar extraction efficiency of 82.5% (55.2g/100g) of potato peel at 135°C after 10min using 10% (w/v) phosphoric acid. Dussan, Silva, Moraes, Arruda & Felipe [12] recorded 71% conversion efficiency using 2% H2SO4 at 155°C in 10min. At 175°C and 2.5% (v/v) phosphoric acid, Orozco, Ahmad, Rooney& Walker [13] recorded 90% glucose conversion from grass. The rate of dilute acid hydrolysis and the yield of simple sugars depend on the biomass type and the process parameters.

Breadfruit husk is one of the agricultural waste whose accumulation at dumpsites and market areas in Nigeria increases because of the current awareness of the incredible food value of breadfruit [14],[15]. Breadfruit husk is about 20% by weight of the dry mass of breadfruit seed and constitutes environmental pollution; traditionally the husk is not used for anything even as an animal feed. This work explored the potential use of breadfruit husk for bioethanol production and the effects of process parameters on the kinetics and thermodynamics of acidic hydrolysis of breadfruit husks.

## 2 MATERIALS AND METHODS

### 2.1 Collection and Preparation of Breadfruit Husk

Breadfruit Husk was collected at a dumpsite at Oye Market Emene in Enugu State of Nigeria. The husk was washed with water to remove sands and other solid impurities. The husk was thereafter sundried for 2 days and crushed with a grinder to get particle sizes below 250µm. The residual moisture was removed by oven drying at 105°C to get the Dried Powdered Husk(DPH)

### 2.2 Characterization of Breadfruit Husk

The water and ethanol soluble extractives were successively determined using the reflux apparatus as described by Sridevi et al. [16]. The total ash content was determined using a muffle furnace as described by Sluiter et al.[17], and Ayeni, Opeyemi, Oyinlola & Temitayo [18]. Both the soluble and the insoluble lignin were determined using the sulfuric acid method [8]. The hemicelluloses after the removal of lignin, were determined by dissolving in potassium hydroxide [17],[18]. The cellulose content was determined by difference after the sequential removal of lignin and hemicelluloses [18].

### 2.3 The Acid Hydrolysis Experiment

The dilute Acid hydrolysis was carried out on the oven-dried husk using hydrochloric acid. Three numeric factors, which are time, temperature, and acid concentration, were considered. The temperature of the hydrolysis reaction was regulated using a hot air oven (DHG-9101-ISA, 75L). The concentrations of the acid used were 0.1M, 0.5M, and 1.0M, which is equivalent to 1%, 5% and 10% sulfuric acid respectively. The experiment was carried out from low to moderately high temperature and because of the wide margin of factor levels, the one-factor-at-a-time (OFAT) method of experiment was deemed necessary for this study. The analysis of simple sugar was carried out using the DNS method [19],[20].

### 2.4 The Kinetic and Thermodynamics Studies

The hydrolysis reaction can be represented as shown in (1) [9] and the accompanied rate equations are shown in (2) and (3).

$$\text{Polymer} \xrightarrow{K1} \text{Monomer} \xrightarrow{K2} \text{Decomposition} \quad (1)$$

$$\frac{dP}{dt} = -K_1[P] \quad (2)$$

$$\frac{dM}{dt} = K_1[P] - K_2[M] \quad (3)$$

Where

K1 and K2 are the rate constants for the formation and decomposition of the simple sugars respectively.

P is the polymer (Cellulose and Hemicellulose) concentrations.

M is the monomer (simple sugar) concentrations.

The equation was developed on the consideration of the reactions as pseudo homogeneous whereas, in principle, the reactions were heterogeneous involving solid phase biomass and liquid phase acid catalysis [21].

The differential equation was combined and solved in terms of the monomer concentration and the result is shown in (4) with the assumption that the biomass as received does not contain any simple sugar [21].

$$M = \left[\frac{K_1 P_0}{K_2 - K_1}\right](e^{-K_1 t} - e^{-K_2 t}) \quad (4)$$

The kinetics equation was solved to obtain the optimum values of K1 and K2 using Microsoft Excel Solver [22]. The target was to minimize the absolute error between the actual data and the model, setting constraints that the values of the rate constants cannot be negative.

Thermodynamic parameters were obtained on the basis that the reactions obey both Arrhenius and Eyring equations [21]. The enthalpy (ΔH) and the entropy (ΔS) changes of the hydrolysis reaction were calculated from the slope and intercept of (5).

$$\ln\frac{K}{T} = -\frac{\Delta H}{RT} + \ln\frac{K_B}{h} + \frac{\Delta S}{R} \quad (5)$$

Where

K= Reaction rate constant (hr-1)

T= Absolute temperature (K)

ΔH= Enthalpy change (J/mol)

R= Gas constant (8.314J/mol.K)

ΔS=Entropy change (J/mol.K)

h = Planks constant (6.626x10-34J.s)

KB= Boltzmann's constant (1.381x10-23 JK-1)

## 3 THE RESULTS AND DISCUSSION

### 3.1 Characterization of the Biomass

The breadfruit husk after sun drying contained about 1% moisture and hence the bulk of the biomass was oven dried before the experiment. The proximate components of the breadfruit are shown in Table 1.

The breadfruit husk contained high amount of non-structural materials that were water and ethanol-soluble also known as the extractives. About 41% of the biomass was extractives, which must first be removed in the analytical procedures to avoid interference with other parameters [23],[24]. Extractives could be non- structural sugars, inorganic materials, nitrogenous materials, chlorophyll, waxes and other minor materials. This amount of extractives was high compared to what was reported on other biomass [16],[25]. The high value of extractives could be attributed to the nonstructural carbohydrates of the breadfruit debris adhered to the husks during processing [26]. The cellulose component was 31% and the hemicelluloses were 15%. Expressing these values in water extractive free basis gave a value of 45% for the cellulose and 22% for the hemicelluloses. These values in extractive free basis fall within the range for most lignocelluloses materials as reported by Yang, Ziyu, Shi-You, & Charles [27]

TABLE 1: THE PROXIMATE COMPONENTS OF BREADFRUIT HUSK

| Parameter | Value (%) |
|---|---|
| Moisture Content | 1.0 |
| Water Soluble Extractives Dry Basis | 32.0 |
| Ethanol Soluble Extractives Dry Basis | 9.0 |
| Acid Soluble Lignin Dry Basis | 9.0 |
| Acid Insoluble Lignin Dry Basis | 4.0 |
| Hemicelluloses Dry Basis | 15.0 |
| Celluloses Dry Basis | 31.0 |
| Ash (%dry Basis) | 9.0 |

### 3.2 The Effects of Process Parameters on the Acidc Hydrolysis of Breadfruit Husks

The effects of time and acid concentration on simple sugar conversion using hydrochloric acid at 70, 90, 150 and 190°C are shown in Fig. 1 to Fig. 4 respectively. At 70°C, the

corresponding differences in sugar conversion with respect to acid concentration were quite significant, and the conversion was proportional to the concentration of acid. The maximum conversion occurred between 20 and 30hrs with 0.1 and 0.5M concentrations of acid, but the maximum conversion with 1.0M occurred earlier. As the temperature was raised to 90°C as can be seen in Fig. 2, the maximum conversion obtained with 0.5M became higher than that of 1.0M, still, the conversion was correspondingly higher than the values at 70°C. However, the time to achieve the maximum conversion still occurred between 20 and 30hrs. When the temperature was increased to 150°C, the maximum conversion with 0.1M jumped by almost 84% of the value at 90°C, but the maximum conversion with 0.5M was still higher at this temperature. It was remarkable that as the temperature was increased to 150°C, the time to achieve the maximum conversion reduced to 2-3hrs. At 190°C, the maximum conversion with 0.1M further increased, but the maximum conversion with 0.5 and 1.0M decreased. In summary, at low temperature, high acid concentration translated to high sugar conversion, but as the temperature increased, the sugar conversion had an inverse relationship with acid concentration. Secondly, the time to achieve maximum conversion decreased with the increase in temperature. Fig. 5 shows the maximum conversion as a function of the acid concentration and temperature, and Figure 3.6 shows the time to achieve maximum conversion as a function of the acid concentration and temperature. Swiatek et al.[28] reported a similar trend where at 200°C the peak glucose concentration during dilute (0.05M) sulfuric acid hydrolysis was achieved in 20min and when the temperature was increased to 220°C, the highest glucose concentration was observed in 10min. Likewise, Megawati et al. [29] reported that the hydrolysis of corn cob using autoclave at 121°C for 1hr with 0.36M sulfuric acid yielded 42.84% simple sugar and the yield increased by 60% as the temperature was increased to 131°C, but the yield dropped to 16.4% when the temperature increased to 160°C. Meanwhile, the time to reach the maximum yield reduced from 1hr at 121°C to 35min at 160°C.

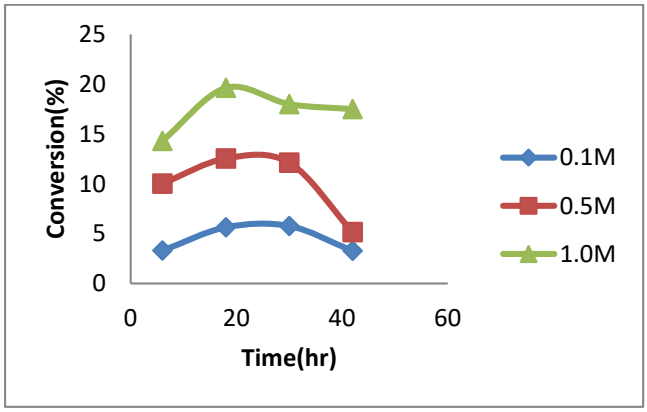


Fig. 1. The effects of acid concentration and time on sugar conversion at 70°C

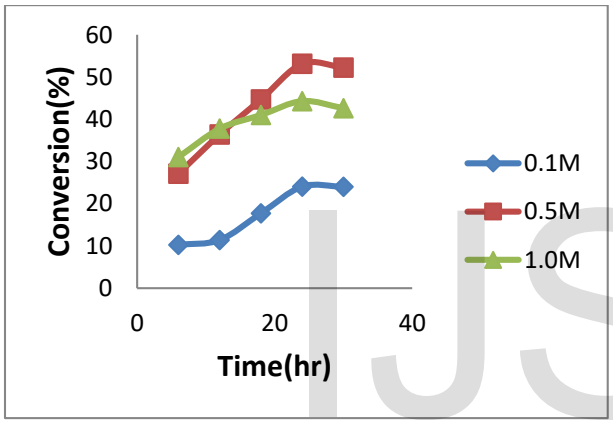


Fig. 2. The effects of acid concentration and time on sugar conversion at 90°C

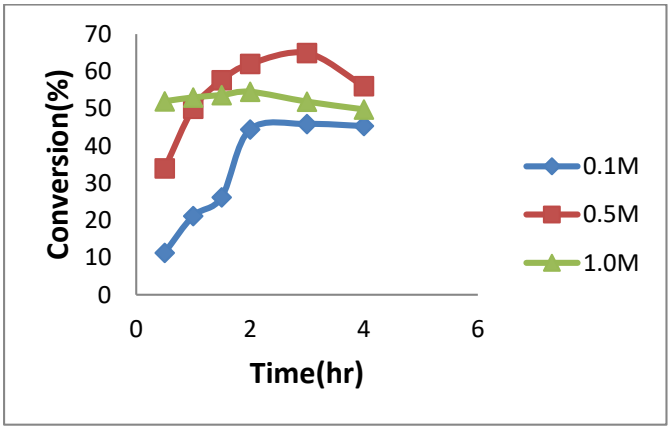


Fig. 3. The effects of acid concentration and time on sugar conversion at 150°C

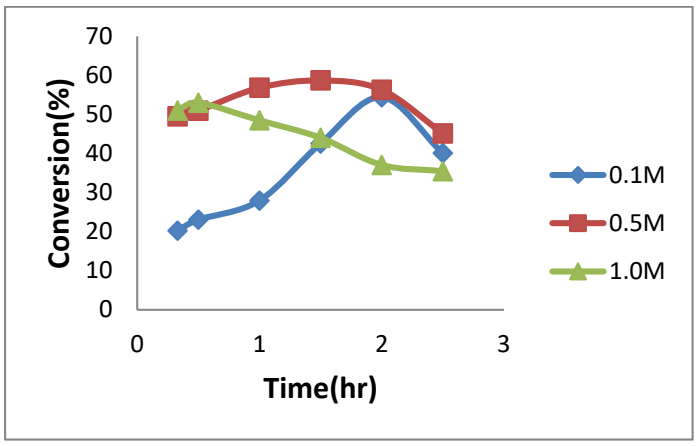


Fig. 4. The effects of acid concentration and time on sugar conversion at 190°C

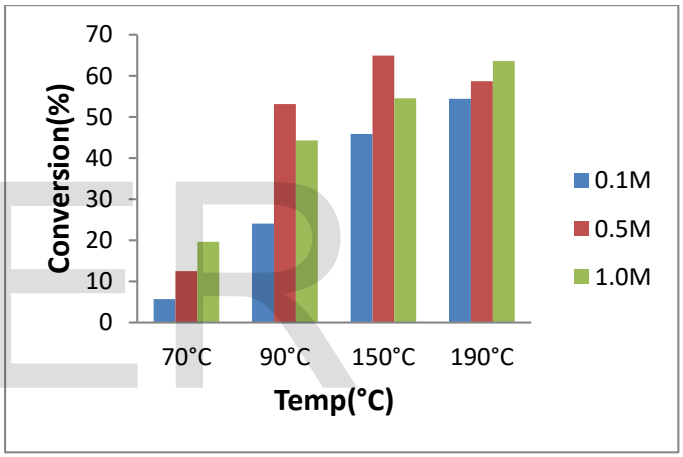


Fig. 5. Maximum Conversion as a function of acid concentration and temperature

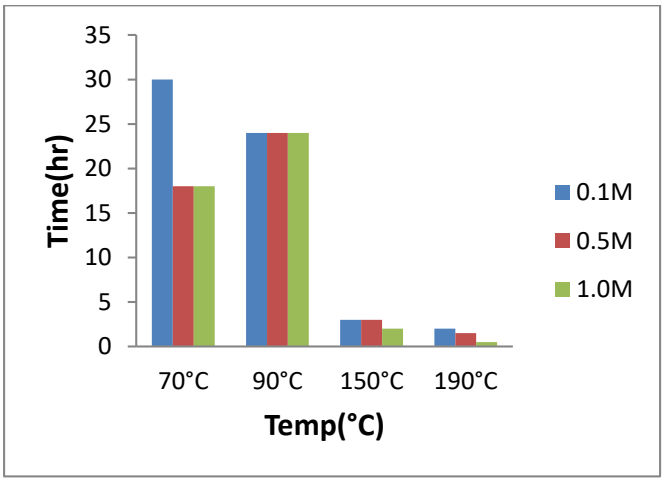


Fig.6. Maximum Conversion time as a function of acid concentration and temperature

### 3.3 The Kinetics

The kinetic parameters are shown in Table 2. Below the temperature of 110°C, the rate of formation K1 and decomposition K2 were slow, meaning that appreciable formation and decomposition of sugars took place above 110°C. Fig. 7 and Fig. 8 show that the rate of formation and decomposition varied proportionally with the acid concentration. Comparing the rate of formation and decomposition, Fig. 3 to Fig.11 show that irrespective of the acid concentration, the rate of decomposition was higher than the rate of formation when the temperature was below 110°C, thereafter, the rate of formation became higher. However, With low acid concentration, the difference between the rate of formation and decomposition at low temperature was higher than that with high acid concentration. The values of the rate constants lie at the same range as what was reported by Belachew &Vijaganand [21]. Orozco et al. [13] however, recorded higher values of rate constants from grass biomass waste using 4% phosphoric acid at 160°C, which can be attributed to the use of high-pressured microwave reactor.

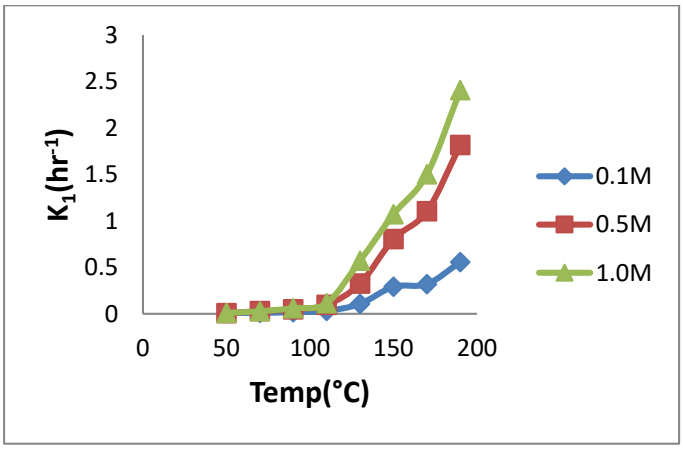


Fig.7. The formation rate constant as a function of temperature and acid concentration

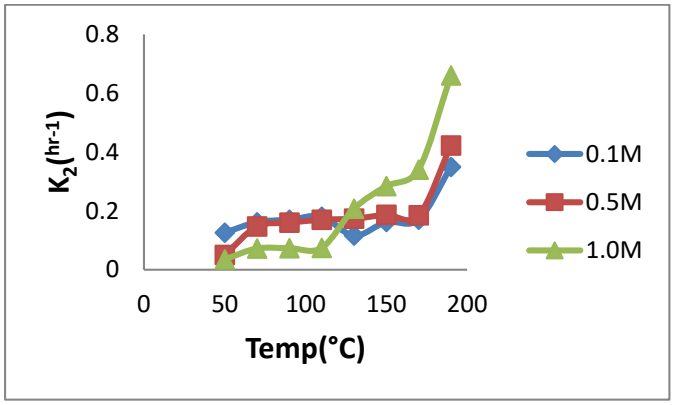


Fig. 8. The decomposition rate constant as a function of temperature and acid concentration

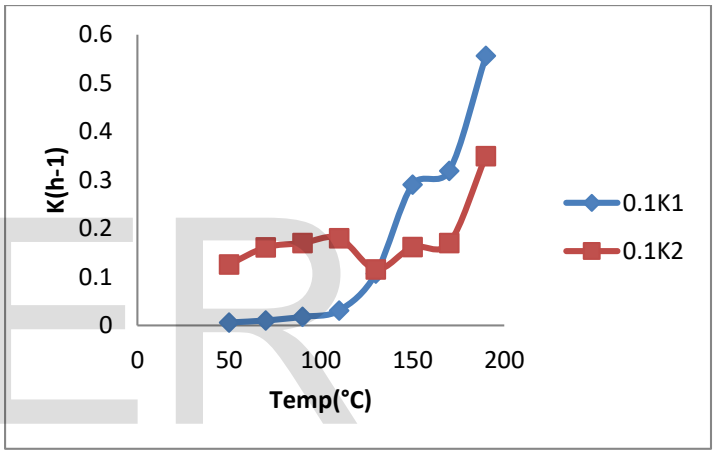


Fig. 9. Formation and decomposition rate constants with 0.1M

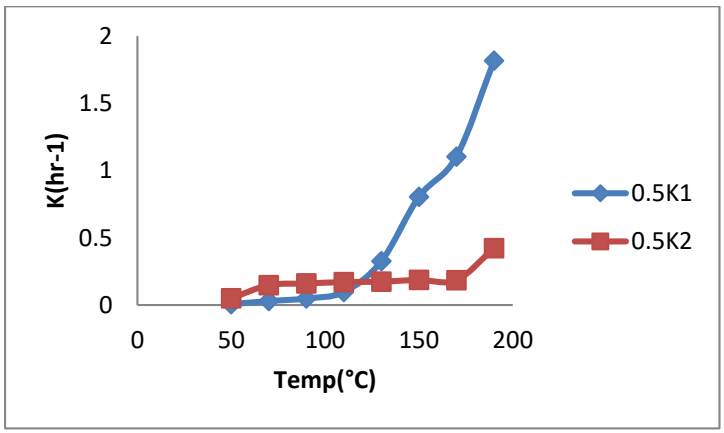


Fig. 10. Formation and decomposition rate constants with 0.5M

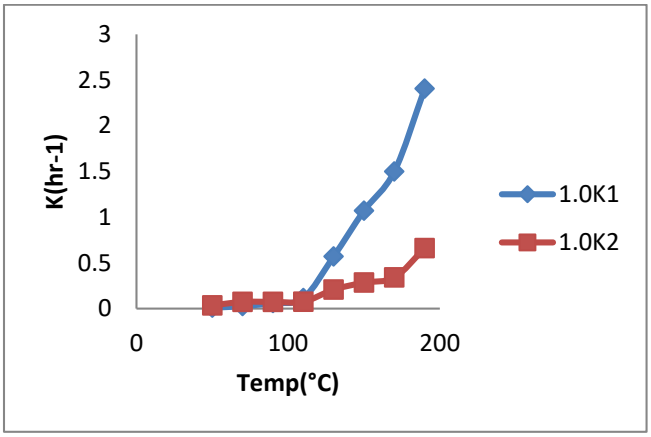


Fig. 11. Formation and decomposition rate constant with 1.0M

### 3.4 The Thermodynamics of the Hydrolyses Processes

The thermodynamic parameters are shown in Table 2. The enthalpy changes are positive showing the process was endothermic. The magnitude of the enthalpy changes was dependent on the acid concentration. The higher the concentration of acid, the higher the enthalpy change. The results indicated that high acid concentration required high energy. The enthalpy changes for the sugar degradation are equally shown in Table 2. The degradation of sugar also required the addition of heat and the magnitude of the enthalpy changes were slightly dependent on the concentration of acid. The data for sugar formation however fitted more into the thermodynamics model than the sugar degradation data. The entropy of the system indicates the stability of the species [13]. Negative entropies of the sugar formation and decomposition indicates that the sugar and the decomposition products were stable. The magnitude of the entropies shows that sugars formed from low acid concentration were more stable than the sugars formed from high acid concentration. Other researchers like Belachew &Vijaganand [21] and Leandro et al. [23] obtained similar result showing positive enthalpy and negative entropy changes.

TABLE 2: THE THERMODYNAMIC PARAMETERS

| Acid Conc(M) | Reaction | ΔH(KJ/mol) | -ΔS(J/mol.K) | R2 |
|---|---|---|---|---|
| 0.1 | Sugar formation | 40.1 | 235.1 | 0.962 |
| | Sugar degradation | 27.7 | 265.4 | 0.982 |
| 0.5 | Sugar formation | 48.4 | 205.1 | 0.983 |
| | Sugar degradation | 35.9 | 246.1 | 0.882 |
| 1.0 | Sugar formation | 47.5 | 205.8 | 0.978 |
| | Sugar degradation | 31.0 | 255.1 | 0.827 |

## 4 CONCLUSION

The breadfruit husks were suitable biomass for bioethanol production with high hydrolysable cellulose and hemicelluloses contents. Dilute concentrations of hydrochloric acid were able to hydrolyze the husk with different conversions depending on the operating condition. At low temperature, the simple sugar yield was directly proportional to the acid concentration. At high temperature, the yield of simple sugars decreased with increase in acid concentration. The kinetics study revealed that the rate of sugar decomposition was higher than the rate of sugar formation at low temperature, but the trend

reversed at temperatures above 110°C. Moreover, the rate of formation and decomposition depended on the acid concentration with high acid concentration giving rise to high rate of formation and decomposition. The thermodynamics analysis showed that both the sugars and the degradation products were stable with negative entropy changes; sugars formed with low acid concentration being more stable than sugars formed with high acid concentration.

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