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Optimization of Acidic Hydrolysis of Sweet Potato Peels To Produce Fermentable Sugar

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Abstract: This study was carried out to optimize fermentable sugar yield via acidic hydrolysis of sweet potato peels. The effects of time, acid concentration, and temperature as process parameters on glucose yield were investigated. The central composite design (CCD) of Response Surface Methodology (RSM) was used and a total of 30 experimental runs were generated for the optimization of the hydrolysis process. A quadratic model equation was obtained for the saccharification of the peels, and the ANOVA test showed the quadratic model obtained to be significant ($p < 0.05$). The results of the model validation for the acid hydrolysis of sweet potato peels were 0.146g/100ml and 0.142g/100ml for predicted and experimented values, with error of about 2.74% established at the optimum conditions of 57°C, 3.41g/50ml substrate concentration, 0.59M acid concentration and 10 hours respectively. The FTIR analysis of the reducing sugar produced at the optimum conditions established shows the presence of aldehyde group, hydroxyl group, alcohol and ketones which confirms it a fermentable sugar.

Keywords: Sweet potato peels, acidic hydrolysis, fermentable sugar, optimization, dilutes sulfuric acid.

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I. INTRODUCTION

Most nations whether economically advanced or at different stages of development are faced with the issue of disposal and treatment of waste (Itelima et al., 2013). These indiscriminate dumping of waste and emissions from fossil fuel has lead to climate change, which causes global warming. The whole world from America to Asia down to Africa are looking for a way to control these pollution menace, thus alternative energy generation is considered one of the options, by converting these wastes (agrowastes) into useful products (Mohammed et al., 2014). These agrowastes are lignocellulose and cellulosic materials. The conversions of cellulose to reducing sugar are usually done through acidic and enzymatic hydrolysis (Kolusheva, 2007). The older method is acidic hydrolysis which requires a high acidic medium (pH 2) obtained through mineral acids; high temperatures (150-230℃) and high pressure (Kolusheva, 2007). As a result of the thermal processing, acidic hydrolysis produces unnecessary by-products which contaminate the end product hydrolysate (Kolusheva, 2007). Dilute acids hydrolysis has been successfully developed for the hydrolysis of lignocellulose materials. Sulphuric acid at concentration usually below 4%wt has been of the most interest in such studies as it is inexpensive and effective. High temperature in the dilute acid treatment is favorable for cellulose hydrolysis (Aiyejagbara, 2015).

The importance of reducing sugar cannot be over emphasized; it is an essential raw material for the production of ethanol, a preferable alternative biofuel in this generation (Mohammed et al., 2014). Agro wastes are generated in large amounts every year, their availability and low cost are characteristics that made them good raw materials for value added products (De-freitas et al., 2009).

Sweet potato botanically known as ipomoea batatas, is a dicotyledonous plant which belongs to the family convolvulaceae, ranked as the seventh most important food crop in global production. In most industries, restaurants and homes where sweet potatoes are eaten or processed into more value-added products, the peels are essentially treated as waste product and they poses disposal problems (Betiku et al., 2013).

In Nigeria and some developed countries, there is a growing interest in the conversion of the huge biomass of organic wastes generated by the food processing sectors into useful products such as fermentable sugar/ethanol (Obianwa et al., 2016). Thus this study was aimed at optimization of fermentable sugar yield via acidic hydrolysis of sweet potato peels.

II. MATERIALS AND METHOD

2.1 PREPARATION OF SWEET POTATO PEELS

Sweet potato peels were obtained from sites where potatoes were fried for sale in Enugu. The peels were washed and dried in an electric oven at 105˚C for 4 hours. The dried peels were ground with an electric grinder, sieved to a mash size of 200µm, and stored in a clean nylon bag at room temperature for acidic hydrolysis.

2.2 PROXIMATE ANALYSIS OF SWEET POTATO PEELS

Kjedhal method was used to analyze the crude protein and sohxlet method was employed to measure fats and oils. The Kurschner-Hanack method (gravimetric method) was used to analyze cellulose and hemicellulose concentrations of the ground peels as employed by Igbokwe et al., (2016); Kulic & Radojicic, (2011).

2.3 PREPARATION OF STANDARD SOLUTION OF H_2SO_4

The concentrations of H_2SO_4 were prepared based on the method in AOAC international 18th edition, 2005.

2.4 ACIDIC HYDROLYSIS OF SWEET POTATO PEELS

50cm^3 each of the prepared concentration of the acid (0.2M, 0.4M, 0.6M, 0.8M and 1M) was added into 250 ml conical flask with varying dosage of (1g, 2g, 3g, 4g and 5g) ground sweet potato peels and heated at an agitation rate of 300 rpm at different temperatures of (30˚C, 40˚C, 50˚C, 60˚C and 70˚C) and different time intervals (1,4,8,12 and 16 hours).

2.5 REDUCING SUGAR DETERMINATION

The DNS method employed by Betiku et al., (2013); Igbokwe et al., (2016) and modified DNS Reagent were used to determine the reducing sugar produced. To 1.0 ml of the hydrolyzed supernatant, 3.0 ml of the DNS solution and 1ml of sodium acetate buffer were added to the test tube, boiled for 15 minutes, cooled and diluted appropriately after which their absorbance were measured at a wavelength of 540 nm using a UV-Visible Spectrophotometer (model).

2.6 OPTIMIZATION OF ACIDIC HYDROLYSIS

Optimization experiment was carried out to determine the optimum process parameters for the acidic hydrolysis of sweet potato peels. The Central Composite Design (CCD) of Response Surface Methodology (RSM) was used for the optimization experiment. Table 2.1 below shows the details of the experimental design.

Table 2.1: Studied range of each factor in coded and actual form for acidic hydrolysis.

Parameter	units	-α	-1	0	+1	+α
Temperature	˚C	40	50	60	70	80
Time	hour	4	8	12	16	20
Substrate dosage	gram	1	2	3	4	5
Acid conc.	Molar	0.2	0.4	0.6	0.8	1.0

III. RESULTS AND DISCUSSION

3.1 PROXIMATE ANALYSIS OF SWEET POTATO PEELS (SPP)

Table 3.1: Percentage composition of proximate analysis of Sweet Potato Peels

Composition	% Composition (dry weight)
Cellulose & Hemicellulose	66
Lignin	11.7
Moisture	9
Ash	4.1
Fats and oil	8.4
Crude protein	0.8

3.2 EFFECT OF PROCESS PARAMETERS ON ACIDIC HYDROLYSIS

3.2.1EFFECT OF ACID CONCENTRATION

The effect of Sulphuric acid concentrations on reducing sugar yield was shown in Figure 3.1. The reducing sugar concentration increases with increase in acid concentration up to 0.6M Sulphuric acid. The highest reducing sugar yield obtained in this study condition of 3g of sweet potato peels hydrolyzed with 0.6M Sulphuric acid at 30˚C for 16 Hrs is 0.141g/100ml. Above 0.6M acid concentration, it was observed that the monomer sugar yields decreased as the acid concentration increased. This could be due to the degradation of product monomers (cellulose and hemicellulose) by acid (Palmarola et al., 2005).

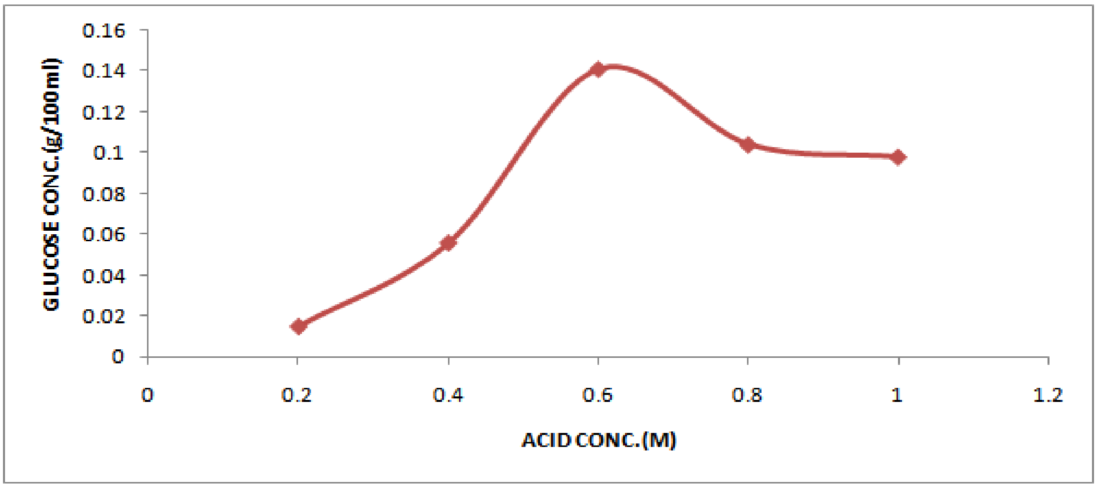

Fig. 3.1: Effect of acid concentration on the yield of fermentable sugar (glucose)

3.2.2 EFFECT OF CONTACT TIME.

The effect of hydrolyzing time on the yield of fermentable sugar was shown in Figure 3.2. At 12 hrs hydrolysis time, maximum reducing sugar yield (0.124g/100ml) was obtained. But after 12 hrs, reducing sugar concentration decreased. These could be as result of decrease in cellulose content of the peel after 12 hrs. The highest glucose yield was recorded at 12 hrs of hydrolyzing 3g of sweet potato peels with 0.6M sulphuric acid at 30˚C. A similar result trend was obtained by Gewchingduang and Pengthemkeerati, (2010).

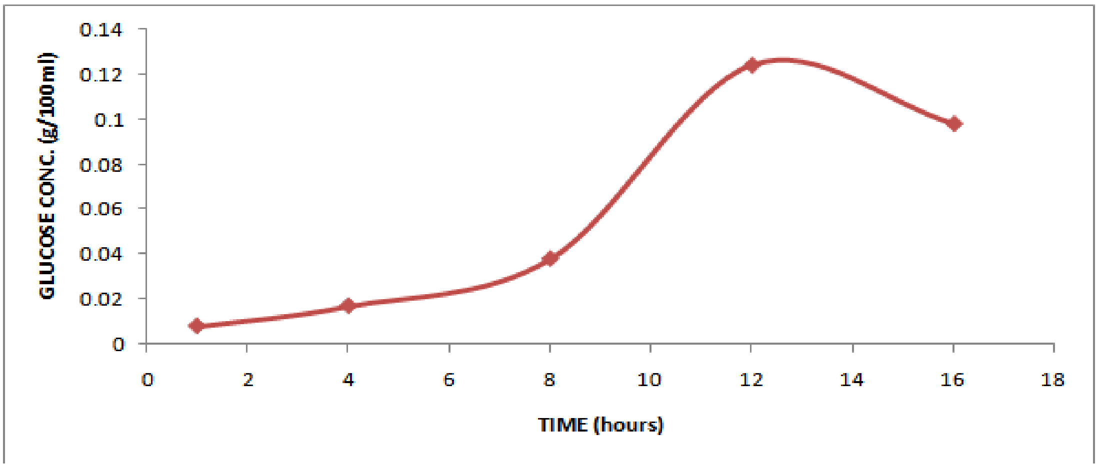

Fig. 3.2: Effect of time on the yield of fermentable sugar (glucose)

3.2.3 EFFECT OF TEMPERATURE.

Figure 3.3 below shows the effect of temperature on the yield of fermentable sugar via hydrolysis of sweet potato peels with 0.6M sulphuric acid. The result showed that increasing temperature had a positive effect on reducing sugar (glucose) yield. Maximum sugar yield of 0.141g/100ml was obtained at 60˚C, after which the yield decreased. A similar observation was reported by Gewchingduang and Pengthemkeerati, (2010). Palmarola et al., (2005) confirmed that higher hydrolysis temperature would enhance the formation of by products that might have adverse effects on hydrolysis process. Hence, hydrolyzing sweet potato peels with sulphuric acid at 60˚C for 12 hrs gave the best reducing sugar yield in this study.

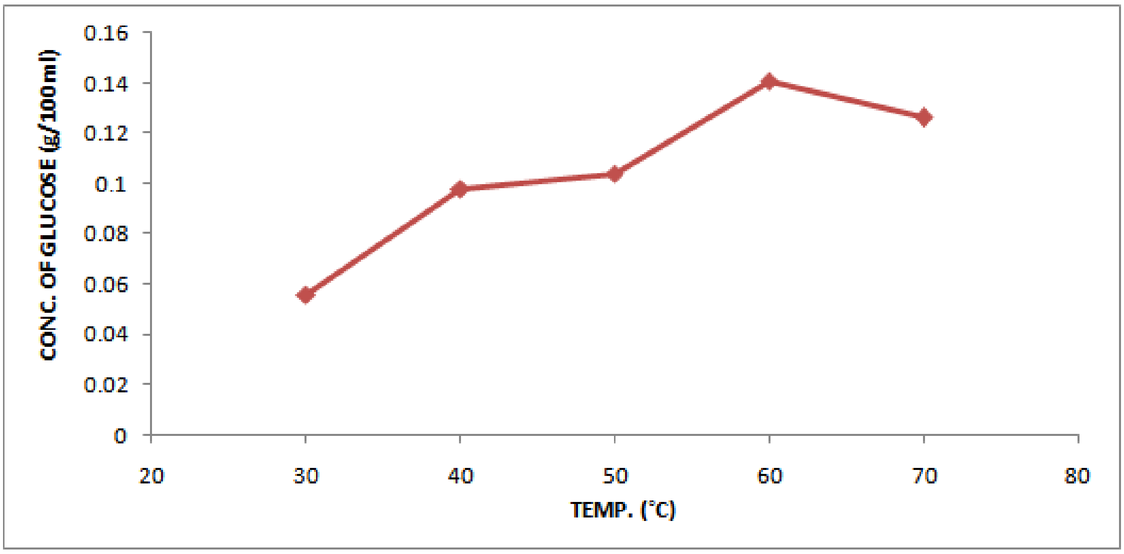

Fig. 33: Effect of temperature on the yield of fermentable sugar (glucose)

3.3 OPTIMIZATION OF GLUCOSE PRODUCTION BY ACID HYDROLYSIS

Experimental design as shown in Table 2.1 was used to optimize the glucose concentration via acid hydrolysis of cellulose. The coded and un-coded values of the test variables were used to optimize the glucose concentration. The concentration of glucose produced depended on the results if there is significant variation for combination of process parameters. The empirical relationship between concentrations of glucose produced (Y) and the four variables in coded values obtained by using the statistical package Design-Expert 6.0.1, version for the determination of the levels of factors which gives optimum glucose concentration was given as the equation below. A quadratic regression equation that fitted the data is:

$$Y = 0.14 - 0.00842A + 0.01B + 0.018C - 0.0005D + 0.00063AB - 0.0078AC + 0.00988AD - 0.01BC + 0.00913BD - 0.003CD - 0.024A^2 - 0.00688B^2 - 0.019C^2 - 0.034D^2 \quad \textbf{(1)}$$

Table 3.2: Significance of regression coefficients of glucose concentration of acid hydrolysis using the design-expert version 6.0.1. version

Source	**Degree of freedom**	**Sum of square**	**Mean Square**	**F-value**	**P-value (Prob >F)**
Model	14	0.064	0.0004538	611.43	< 0.0001
A (Temp.)	1	0.0017	0.0017	229.06	< 0.0001
B (Time)	1	0.002563	0.002563	345.27	< 0.0001
C (Substate dosage)	1	0.007561	0.007561	1018.76	< 0.0001
D (Acid conc.)	1	0.000006	0.000006	0.81	0.3828
AB	1	0.00000625	0.00000625	0.84	0.3733
AC	1	0.000961	0.000961	129.48	< 0.0001
AD	1	0.00156	0.00156	210.21	< 0.0001
BC	1	0.001681	0.001681	226.48	< 0.0001
BD	1	0.001332	0.001332	179.49	< 0.0001
CD	1	0.000144	0.000144	19.40	0.0005
A^2	1	0.015	0.015	2084.47	< 0.0001
B^2	1	0.001296	0.001296	174.67	< 0.0001
C^2	1	0.01	0.01	1351.67	<0.0001
D^2	1	0.032	0.032	4303.43	< 0.0001
Residual	**15**	**0.0001113**	0.000007422		
Cor. Total	**29**	**0.064**			

Std. Dev. = 0.00272; Mean = 0.076; C.V.% = 3.58; PRESS = 0.000551; R^2 = 0.9983; Adj. R^2 = 0.9966; Pred. R^2 = 0.9913; Adeq. Precision = 72.673

Table 3.3: Saccharification data for the actual glucose concentration, predicted glucose concentration and residual values

Standard order	**Predicted values(g/100ml)**	**Actual/Observed values(g/100ml)**	**Residual**
10	0.003	0.003	-0.0
24	0.006	0.004	-0.002
23	0.008	0.006	-0.002
1	0.019	0.018	-0.001
9	0.026	0.025	-0.001
18	0.031	0.031	-0.000
21	0.031	0.029	-0.002
2	0.036	0.036	-0.000
14	0.038	0.037	-0.001
3	0.041	0.042	+0.001
16	0.057	0.061	+0.004
4	0.060	0.062	+0.002
12	0.064	0.065	+0.001
17	0.065	0.064	-0.001
8	0.066	0.067	+0.001
7	0.077	0.080	+0.003
6	0.083	0.084	+0.001
11	0.084	0.085	+0.001
13	0.091	0.092	+0.001
19	0.095	0.097	+0.002
5	0.097	0.096	-0.001
22	0.102	0.099	-0.003
15	0.108	0.109	+0.001
20	0.136	0.131	-0.005
26	0.143	0.141	-0.002
27	0.143	0.145	+0.002
30	0.143	0.141	-0.002
25	0.143	0.145	+0.002
28	0.143	0.142	-0.001
29	0.143	0.145	+0.002

The glucose concentration was optimized with the design expert software (CAD) giving a maximum yield of 0.146g/100ml H_2O at optimum conditions; 57°C, 10 hours, 3.41g/50ml substrate dosage and 0.59M acid concentration with desirability of 1.000. Acidic hydrolysis of the peel under the observed optimum operating conditions was carried out in order to evaluate the precision of the quadratic model; the experimental value and predicted values are shown in Table 3.4. Comparing the experimental and predicted results, it can be observed that the error between the experimental and predicted is less than 3%, therefore it can be concluded that the generated model has sufficient accurancy to predict the glucose production via acid hydrolysis.

Table 3.4: Results of the model validation (experiment to validate the optimum glucose production by acid hydrolysis of cellulose in sweet potato peels).

Temp $^{\circ}$C **A**	Time (Hrs) **B**	Substrate dosage (g/50ml) **C**	Acid concentration (Molar) **D**	Experimented Glucose concentration (g/100ml H_2O)	Predicted Glucose concentration (g/100ml H_2O)	% Error
57	10	3.41	0.59	0.142	0.146	2.74

Figure 3.4: FTIR spectral analysis of fermentable sugar produced from sweet potato peels at the optimum conditions.

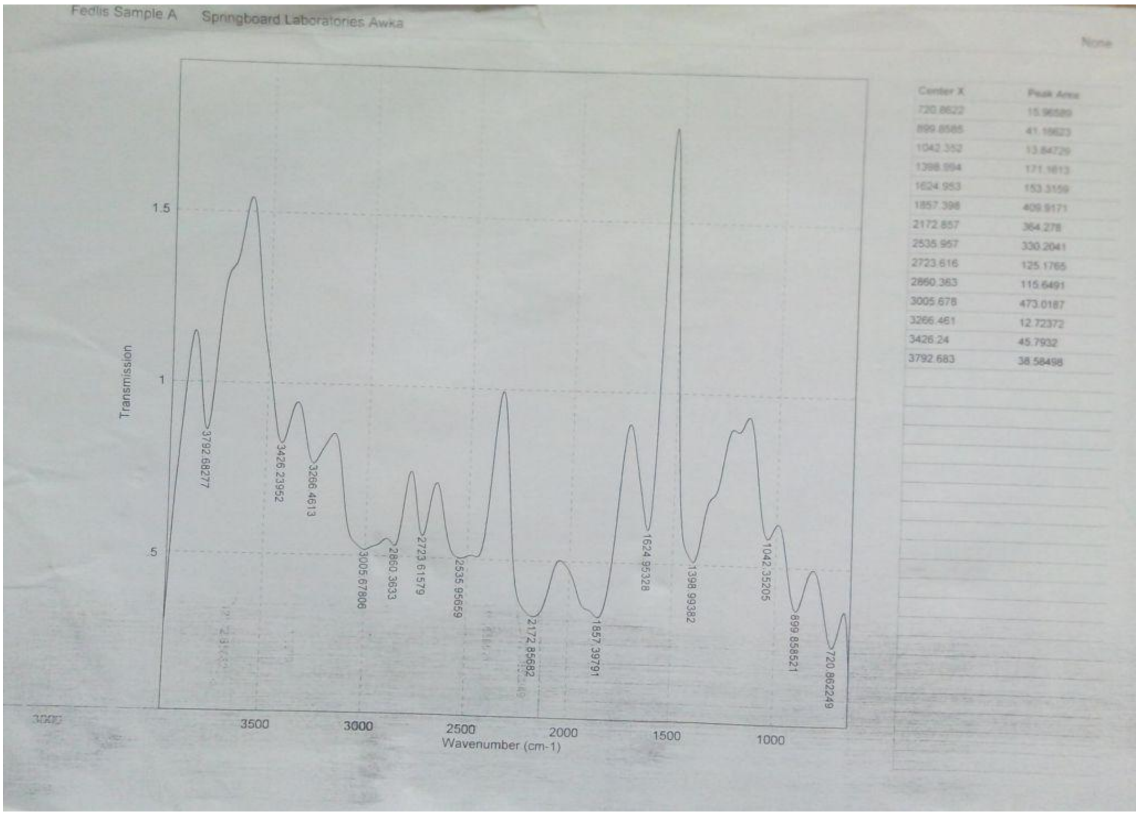

Table 5: Fourier Transform Infrared (FTIR) Spectrum of fermentable sugar from sweet potato peels.

Wave number (cm^{-1})	Bond source (functional group)
720.86	C–H, out of bend aromatics eg alkanes, C–CL, Flourides
899.85	C –H, out of bend aromatic
1042.3	C –O, alcohols, esters, ethers, carboxylic acid & anhydrides.
1398.9	C–F, Flouride
1624.9	N–H, bend in amide & C=C, alkene.
1857.3	C–H, bend of aromatic compound
2172.8	C≡ alkyne group, S–C≡N (thiocyanate).
2535.9	O–H, carboxylic acids
2723.6	C–H, aldehydes.
2860.3	O–H, alcohols, carboxylic acid & N–H, amine salt.
3005.6	C–H, stretch of alkanes
3266.4	O–H, H-bonded alcohols & phenols
3426.2	N–H, stretch of primary amines and amide.
3792.6	N–H, stretch of amide.

The wave lengths from the spectrum as indicated above shows the functional groups present in the reducing sugar produced.

IV. CONCLUSION

The quadratic model generated from the optimization of acidic hydrolysis of sweet potato peels using central composite design (CCD) of Response surface methodology (RSM) was adequate in predicting the fermentable sugar yield. Temperature, time, acid concentration and substrate dosage were significant factors that affected the yield of fermentable sugar in acidic hydrolysis. The optimum parameters established were 57˚C, 10 hrs, 0.59M and 3.41 g/50ml for temperature, time, acid concentration and substrate concentration respectively. The FTIR analysis confirmed the presence of the basic functional groups of fermentable sugar.

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