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Pelagia Research Library

Der Chemica Sinica, 2013, 4(1):67-74

Der Chemica Sinica
Pelagia Research Library
ISSN: 0976-8505
CODEN (USA) CSHIA5

Analysis of Abakaliki Rice Husks

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ABSTRACT

In Nigeria, rice is grown all over the country but Abakiliki zone of Ebonyi state is the largest producer, giving rise to tonnes of rice husks which are indiscriminately dumped around the rice mills. Hence allowing uncontrolled degradation and fermentation where they increase the biological oxygen demand of streams in which they are dumped. This aroused the interest in research into the use of this abundant biomass. The proximate analysis of Abakaliki rice husks was carried out, the results obtained compared favourably well with that of literature values.

Key words: Rice husks and proximate analysis

INTRODUCTION

Ideas for alternatives to ever decreasing reserves of fossil fuels as well as measures to reduce the CO_2 emissions have been the centre for international concerns and discuss in the face of global warming. However, the efficient management of biomass to produce bio fuels is one of the most interesting aspects under investigation in order to achieve environmentally – clean and CO_2 neutral solution[1] Daniel Schneider, Marina Escala, Kawin Supawittayayothin, Nakorn Tippayawong, *on the characterization of biochar from hydrothermal carbonization of bamboo*;2011:International journal of energy and environment vol. 2, 4, 647-652. Lignocellulosic biomass, such as agriculture waste, wood, grass and bamboo are renewable resources that do not divert feed stock from food streams[2] Zheng Y, Zhongli P, Zhang R; *overview of biomass pre-treatment for cellulosic ethanol production*; 2009; Int. J. Agric. & Biol. Eng. 2, 51 – 68, but rather used in producing alcohols and other volatile compounds.

In most developing countries of the tropics, carbohydrate-based agricultural commodities such as starchy tubers and cereals occur abundantly. The high cost of substrates has been identified as a major factor affecting the economic viability of acetone and butanol fermentation. This and the ability of the saccharolytic organism to utilize many different carbohydrates have aroused interest in research into the use of alternative and cheaper substrates. These cheaper substrates can be seen in the agro-waste being dumped indiscriminately and which are abundant in nature. This careless dumping creates attendant waste disposal of these waste rather than allowing them to undergo uncontrolled degradation and fermentation at dump sites where they increase the biological oxygen demand of streams in which they are dumped [3] Fatile I. A., Layokun S.k. and Solomon B.O; *Lactase Fermentation by Yeasts: Candida Pseudotropicals and candida Kyfer;* 1988; Journal of Mig. Soc. of Chem. Engineers.

The analysis of Abakaliki rice husks is necessitated as a part of numerous work to the production of bio fuel using cellulose as one of the most abundant renewable biomass on earth. Cellulose accounts for about 50% of the total agricultural, cellulose rich residues. However, several other plants are useful candidates to provide biomass for the production of ethanol. Cellulose in biomass is hydrolysed to glucose by acid hydrolysis than hemicelluloses. The reducing sugar obtained can be fermented to give ethanol.

The use of rice husks cannot be over emphasized, it ranges from industrial to agricultural uses, and recently to alcohol production.

MATERIALS AND METHODS

50g sample material was milled for 34hrs; air dried and separated into particle sizes 425µm and 300µm, and was used for the determination of the following:

(i) *Moisture content*
Apparatus: Drying oven, analytical weighing balance, desiccator and watch glasses.
Material: 1g of 300µm sample of Abakaliki rice husk

Procedure
1g test specimen was placed in the watch glass that has been previously washed, dried and weighed. The watch glass plus the specimen were placed in the drying oven at 105^0C, with the watch glass uncovered for one hour. Then it was replaced, cooled in a desiccator and weighed. The drying and weighing procedure were repeated until a constant weight was obtained. [4] American Standard Test Method D 2974 – 87

(ii) *Ash content*
Apparatus: Crucibles, muffle furnace , analytical weighing balance , drying oven.
Material: 2g of 300µm sample of Abakaliki rice husks.

Procedure
Empty crucible was washed, dried and ignited in a muffle furnace at 600^oc. This was cooled in a dessicator and weighed. 2g of the test specimen was then placed in the crucible. The weight of the crucible plus the specimen was determined and placed in an oven at 105^0C for 1hr uncovered. The cover was replaced and cooled (to prevent absorption of moisture from the air) in desiccator and weighed. The cooled specimen was placed in the muffle furnace uncovered and was ignited at 600^0C until all the carbon was eliminated. After ignition, it was later placed in a desiccator , cooled and weighed.

The heating process was repeated at 30min interval until a constant weight was achieved. [4] American Standard Test Method D 2974 – 87

(iii) *Fat content*
Apparatus: Soxhlet Extraction apparatus, Hot plate, Condenser and Filter paper
Material: 2g of 300µm sample of Abakaliki rice husks
Reagent: Hexane or petroleum ether

Procedure
2g of air dried sample placed in soxhlet extraction apparatus. The flask was filled to (2/3) of its volume with solvent (Hexane) and was used for the continuous extraction for 6 hours. At the end of the extraction, the solvent was distilled off, leaving the extract in the flask. The extract with the flask was dried in an oven at 40^0C to remove all residual solvent, cooled in a desiccator and weighed. [5] Association of Official Analytical Chemists(1975),12th Edition.

(iv) *Crude fibre*
Apparatus: Flasks, Filter paper, Oven, Crucible and Muffle furnace
Material: 2g of 300um sample of Abakiliki rice husks
Reagent: Diethyl ether, 1.25% H_2S0_4, 1.25% NaOH

Procedure
2g weight of the test specimen was defatted with diethyl ether for 8hrs. This was boiled with 200ml of 1.25% H_2S0_4 under reflux for 30min. The washed material was boiled with NaOH for exactly 30min and filtered, after filtration it was dried in an oven at 100^0C, cooled and weighed.

The material was burnt in a muffle furnace at 600^0C for 2 ½ hrs, cooled and weighed.

(v) *Protein content*
Apparatus: Flask, Hot plate and Distillation Unit
Materials**:** 1g of 300um rice husks
Reagents: Sodium hydroxide 70% w/v, Copper sulphate ,Boric acid 4% w/v, Sulphuric acid and Sodium sulphate

Procedure
1g of the sample was added into a known weight filter paper, wrapped and dropped into a flask. 20ml of conc. Sulphuric acid, 5g of sodium sulphate and 1g of copper sulphate were added to the content of the flask. The flask was heated in a fume cupboard with the temperature gradually raised to 750^0c. The digestion was carried out for 12hr. After, the digest was dissolved with 20ml of distilled water and poured into a 500ml flat bottom flask. 50ml of diluted 70% sodium hydroxide solution was gradually poured to the flask and the apparatus set for distillation. 50ml of 4% Boric acid with two drops of methyl red indicator was put in a 250ml conical flask which is the receiver of the distillation unit. The apparatus was heated to distil off ammonia. The distilled ammonia was absorbed by boric acid till 150ml of the distillate was collected. Then, the ammonia was estimated by titrating it with 0.1N sulphuric acid with a colour change of blue to red observed.

(vi) *Cold water solubility*
Apparatus: Filtering crucible and Suction Filtering flask
Material**:** 2g 300µm sample of Abakiliki rice husks.

Procedure
2g sample was placed in a 400ml beaker and covered with 300ml of distilled water. The mixture was allowed to digest at a temperature of 25^oc with frequent stirring for 48 hours. It was filtered and the residue was dried in an oven and weighed after cooling.

(vii) *Hot water solubility*
Apparatus: Flask, Reflux condenser and Water bath or kettle boiler
Material: 2g test specimen of 300µm sample of Abakiliki rice husks.

Procedure
A 2g test specimen, whose moisture content had been previously determined, was placed in a conical flask and 100ml of distilled water added. This was placed in a boiling water bath with the solution in the flask just below the level of the water in the bath. A reflux condenser was attached to the flask and was heated for three hours. The contents of the flask were filtered using filter paper, washed with hot water and dried to constant weight at 105^0C[6] American Standard Test Method D 1110-56
.
(viii) *Alcohol –toluene solubility*
Apparatus: Filtering crucibles, Soxhlet Extraction apparatus and Condenser
Material: 2g of 3ooum sample of Abakiliki rice husks
Reagent: Alcohol –Toluene solution obtained by mixing 1 volume of ethyl alcohol (95 %) and 2 volumes of chemically pure toluene.

Procedure
2g weight of the test specimen was placed in a soxhlet extraction apparatus. The test specimen was extracted with 150ml of alcohol –toluene solution for 6hr, while keeping the liquid boiling briskly. At the end of the extraction, the solvent was distilled off. The flask with the extract was dried in an oven at 105^0C for 1hr, cooled in a desiccator and weighed [7] American Standard Test Method D 1107-56
.
(ix) *Acid soluble lignin*
Apparatus
1. Spectrophotometer, suitable for measuring absorbance at the ultra violet range of radiation.
2. Absorption cells (cuvettes), fused silica, with 10mm light path.

Material: Filtrate obtained from decanting the supernatant solution after settling of acid insoluble lignin.
Reagent: 3% Sulphuric acid

Procedure
The absorbance was measured on a test specimen at 205mm in a cuvette with 10mm light path. 3% sulphuric acid was used as a blank on reference solution. For the absorbance higher than 0.7 the filtrate was diluted with 3% sulphuric acid to obtain absorbance in the range of 0.2 -0.7 and diluted filterable used as test specimen.

(x) *Acid insoluble lignin*
Apparatus: Filtering crucible, Water bath, Reflux condenser, Drying oven, Hot plate and Flasks
Material: 2g of 300um sample of Abakiliki rice husks
Reagents: 2% Sulphuric Acid solution

Procedure
2g of the sample was put into a beaker, 15cm^3 of 72% sulphuric acid cooled to 15^{0}C was gradually added while stirring and macerating the material with glass rod. The beaker was kept in a water bath maintained at room temperature 30^{0}C for 2hrs, stirring the material frequently during this time.

The content of the beaker was transferred into a 350ml of water in a conical flask. The beaker was rinsed with water diluting to 3% sulphuric acid for a total volume of 575cm^3. The solution was boiled for 4hrs maintaining constant volume by using a reflux condenser.

The insoluble material (lignin) was allowed to settle, keeping the flask in an inclined position overnight. The supernatant solution was decanted carefully. The lignin was filtered and washed free off acid with hot water. The residue was dried in an oven at 105^{0}C to constant weight, cooled in a desiccator and weighed.

RESULTS AND DISCUSSION

The results obtained from the proximate analysis of the rice husks compared favourably well with that of the literature values. There were no significant variations except for the protein, ash and fat content analysis. This could be attributed to the fact that some inorganic materials dissolved in the excess moisture, therefore resulting to high ash, protein and fat content analysis of Abakaliki rice husk.

Table 1: Proximate analysis of Abakaliki rice husk

	%Moisture	%Ash	%Fat	%Crude fibre	%Protein	%Carbohydrate
Abakaliki rice husk	9.93	25.82	0.49	29.09	2.51	28.19
Literature value	12.0	24.0	0.30	30.0	1.0	30.0

Extractives
Extractives in rice husk consist of materials that are soluble in neutral solvents and that are part of the rice husk. They are very important parameters in determination of acid insoluble lignin, and as such must be removed from the rice husk before estimating for acid-insoluble lignin because of their interference with the result.

Table VI: Values of the extractives

	%Alcohol-Toluene solubility	%Cold water solubility	%Hot water solubility
Abakaliki rice husk	3.97	15.87	15.39
Literature value	3.00	--	--

CONCLUSION

The proximate analysis of Abakaliki rice husks was carried out, the results obtained compared favourably well with that of literature values.

REFERENCES

[1] Daniel Schneider, Marina Escala, Kawin Supawittayayothin, Nakorn Tippayawong, **2011**:*International journal of energy and environment* vol. 2, 4, 647-652.
[2] Zheng Y, Zhongli P, Zhang R; **2009**; *Int. J. Agric. & Biol. Eng*. 2, 51 – 68
[3] Fatile I. A., Layokun S.k. and Solomon B.O; *Lactase Fermentation by Yeasts: Candida Pseudotropicals and candida Kyfer;* **1988**; *Journal of Mig. Soc. of Chem. Engineers.*
[4] American Standard Test Method D 2974 – 87
[5] Association of Official Analytical Chemists (**1975**), 12th Edition.
[6] American Standard Test Method D 1110-56
[7] American Standard Test Method D 1107-56

APPENDICES

APPENDIX I: MOISTURE DETERMINATION

	A (g)	B (g)
Weight of sample	1.0018	1.0116
Weight of dish + sample	16.8952	16.9570
Weight of dish + residue	16.7951	16.8573
Loss in weight	0.1001	0.0997

% Moisture $= \frac{W1\ X\ 100}{w2}$

Where W_1 = weight of sample

W_2 = loss in weight after oven dry

A $= {0.1001\ x\ 100}/{1.0018} = 9.99\%$

B $= {0.0997\ x\ 100}/{1.0116} = 9.86\%$

% Moisture $= {9.99 + 9.86}/{2} = 9.93\%$

APPENDIX II: ASH DETERMINATION

READINGS		
	A (g)	B(g)
Wt. of sample	1.9981	2.0000
Wt. of ash	0.4561	0.4738

%Ash $= \frac{W1\ X\ 100}{W2P}$

Where: W_1 = weight of ash

W_2 = weight o oven –dry sample

P = proportion of moisture free rice husk in air-dry specimen

% Ash_1 $= {0.4561\ x\ 100}/{1.9981 - (0.0993\ x\ 1.9981)} = 25.34\%$

% Ash_2 $= {0.4738\ x\ 100}/{2.0000 - (0.0993\ x\ 2.0000)} = 26.30\%$

% Ash $= {25.34 + 26.30}\ /2\ \ 0 = 25.82\%$

APPENDIX III: FAT DETERMINATION

READINGS		
	A (g)	B(g)
Wt. of Sample	2.0051	1.9998
Wt. of flask	247.9511	248.0011
Wt. of flask + extract	247.9605	248.0112
Wt. extract	0.0094	0.0101

% Fat $= \frac{W3-W2\ \ X\ (100)}{W1}$

W_1 = weight of sample

W_2 = weight of empty flask

W_3 = weight of flask + oil

% Fat_1 = ${0.0094\ X\ 100}/{2.0051} = 0.47\%$

% Fat_2 = ${0.0101\ \ x\ \ 100}/{1.9998} = 0.51\%$

% Fat = ${0.47\ + 0.51}/{2} = \ 0.49\%$

APPENDIX IV: CRUDE FIBRE DETERMINATION

READINGS		
	A (g)	B (g)
Weight of sample	2.0000	2.0051
Weight after oven dry	1.7600	1.7523
Weight after furnace burning	1.1374	1.2101

% FIBRE = $\frac{(C2-C3)X\ 100}{C1}$

Where: C1 = weight of the original sample

C_2 = Weight after oven dry

C_3 = weight after furnace burning

% Crude $Fibre_1$ = $\frac{(1.7600-1.1374)X\ 100}{2.0000}$

= 31.13%

% Crude $Fibre_2$ = $\frac{(1.7523-1.2101)X\ 100}{2.0051}$

= 27.04%

% Crude Fibre = $\frac{31.13+27.04}{2}$

= 29.09%

APPENDIX V: PROTEIN DETERMINATION

READINGS		
	1	2
Wt. of Sample (g)	0.9998	1.0012
Titre (ml)	2.75	2.97

1ml of 0.1N H_2SO_4 = 0.0014gN

%Protein = $\frac{V\ x\ 0.0014\ x100\ x6.25}{W}$

W = Weight of sample

V = volume of acid

6.25 = empirical factor

% $Protein_1$ = $\frac{2.75\ x\ 0.0014\ x100\ x6.25}{0.9998}$

=2.41%

% $Protein_2$ = $\frac{2.97x\ 0.0014\ x100\ x6.25}{1.0012}$

= 2.60%

% Protein = $\frac{2.41+2.60}{2}$ = 2.51%

APPENDIX VI: ALCOHOL – TOLUENE SOLUBILITY

READINGS		
	1 (g)	2 (g)
Wt. of Sample	2.0000	2.0150
Wt. of flask	249.1000	248.2500
Wt. of flask + extract	249.1728	248.3208
Wt. extract	0.0728	0.0708

% Alcohol – Toluene soluble component = $\frac{W_1 \text{ x } 100}{W_2 p}$

W_1 = weight of dried extract

W_2 = weight of sample

P = Proportion of moisture free rice husk in air – dry specimen

% Alcohol – Toluene $solubility_1$ = $\frac{0.0728x\ 100}{2-(2\ x\ 0.0993)}$

= 4.04%

% Alcohol – Toluene $solubility_2$ = $\frac{0.0708\ x\ 100}{2.015-(2.015\ x\ 0.0993)}$

= 3.90%

% Alcohol – Toluene solubility = $\frac{4.04 + 3.90}{2}$

= 3.97%

APPENDIX VII: CARBOHYDRATE DETERMINATION

Carbohydrate = 100 – (% ash + % moisture + % fat + %Protein + %Crude fibre + %extractives)

Carbohydrate = 100 – (9.93% + 25.82% + 0.49% + 29.09% + 2.51% + 3.97)

= 28.19%

APPENDIX VIII: COLD WATER SOLUBILITY DETERMINATION

READINGS		
	A(g)	B (g)
Wt. of Sample	2.0010	2.0000
Wt. of Residue	1.5004	1.5314

% cold water solubility = $\frac{(W1-W2)X\ 100}{W1}$

W_1 = weight of moisture –free specimen

W_2 = Weight of dried specimen after extraction with cold water

% cold water solubility$_1$ = $\frac{(1.8023 - 1.5004)}{1.8023} \times 100$

=16.75%

% cold water solubility$_2$ = $\frac{(1.8014 - 1.5314)}{1.8014} \times 100$

= 14.99%

% cold water solubility = $\frac{16.75 + 14.99}{2}$

= 15.87%

APPENDIX IX: HOT WATER SOLUBILITY DETERMINATION

READINGS		
	A (g)	B (g)
Wt. of Residue	1.5290	1.5352
Wt. of Sample	2.0000	2.0121

% Hot water solubility = $\frac{W_1 - W_2 \times 100}{W_1}$

W_1 = Wt. of moisture free sample

W_2 = Wt. of residue

% Hot Water solubility$_1$ = $\frac{(1.8014 - 1.5290) \times 100}{1.8014}$

= 15.12%

% Hot water solubility$_2$ = $\frac{(1.8123 - 1.5285) \times 100}{1.8123}$

= 15.66%

% Hot water solubility = $\frac{15.12 + 15.66}{2}$

= 15.39%

APPENDIX X: ACID – SOLUBLE LIGNIN DETERMINATION

Lignin content B in the filtrate in grams per 1000cm3

B = A/W

A = Absorbance (0.885)

B = lignin content in the filtrate in g/1000cm3

V = Total volume of filtrate

W = oven dry weight of husks in gms

Wt. of Oven dry sample = 1.938

B = A/W = $0.885/1.938$ = 0.457

% acid soluble lignin = $\frac{B\ X\ V\ X100}{1000\ X\ W}$

= $\frac{0.457 \times 575}{10 \times 1.938}$

= 13.56%

APPENDIX XI: DETERMINATION OF ACID INSOLUBLE LIGNIN

READINGS		
	A (g)	B (g)
Residue	0.4628	0.5089
Wt. of oven dry sample	1.9380	1.9380

% Lignin = $A\ x\ 100/W$

Where: A = Weight of lignin (g)

W = Oven dry weight of test specimen (g)

$$\% \text{ acid insoluble lignin}_1 = \frac{0.4628 \times 100}{1.9380}$$

$$= 23.88\%$$

$$\% \text{ acid insoluble lignin}_2 = \frac{0.5089 \times 100}{1.9380}$$

$$= 26.26\%$$

$$\% \text{ acid – insoluble lignin} = \frac{23.88 + 26.26}{2}$$

$$= 25.07\%$$

$$\% \text{ Actual acid insoluble Lignin} = 25.07 - (15.39 + 3.97)$$

$$= 5.71\%$$

% Lignin = % acid insoluble + % acid soluble

= 5.71 + 13.56

= 19.27%