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Preparation and Characterization of Maize Tassel Fibre for Adsorption Processes

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ABSTRACT

In this study, the surface physical characterization, chemistry characterization and proximate analysis were carried out on maize tassel fibres in order to check the feasibility of the bio-sorbent for bio-sorption process. The moisture content, ash content, apparent density, particle size and crude protein results of maize tassel fiber were carried out and the values gotten were 4.51 %, 2.134 %, 0.41 g/ml, 300 μm and 4.06 % respectively. The low amount of the aforementioned properties shows that the bio-material is exceptional for the use of column adsorption. The surface chemistry of the bio-material shows the presence of carboxylic groups and lactonic groups present in the material but the presence of phenolic groups is minimally present; this was shown using Fourier Transform- Infrared spectroscopy and Boehm titration showed the number of acidic sites present in the material to be 0.9 mmol/g. The Brunauer–Emmett–Teller (BET) isotherm was used to find the surface area, pore volume and pore size diameter to be 652.3 m^2/g , 0.4056 cm^3/g and 2.144 nm respectively. The Iodine number for the sorbent material was 423.049 which are within the standard range of a standard adsorbent. The results indicated that maize tassel fibre is mesoporous and a good material for biosorption process.

Keywords: BET, FT-IR, Maize tassel fibre

1 INTRODUCTION

Maize tassel (known as the male flower) is found at topmost part of the plant which produces pollen grains transferred by current of air to fertilize the female flowers called the silk. One maize tassel fibre can produce in excess an average of 25 million pollen grains to fertilize the ear of the corn which are useful between 1- 4 hr which would detach and end up covering a distance of 300 m, while the cob produces not less than 1000 filled seeds. During seed production, one line of the tassel fibre that produces pollen grains is sufficient to fertilize 2–3 lines of the female flower in the following proportion of 1:4 or 1:6 (male: female). Thus, maize tassel as a natural occurring matter can be utilized to yield monetary worth of products due to the presence of bioactive compounds (Duangpapeng *et al.*, 2018).

The growth cycle of maize is about three to four weeks after fertilization. During the process of fertilization the maize tassel plays an important role. This implies that beyond fertilization the tassel has no use to the farmer. The farmer cuts it off after fertilization to ease the part that will yield viable kernels (Mamba *et al.*, 2012).

The maize used in this work is known as (*Zea mays*) which is one of the essential edible grains

globally. The by-products of maize plant which are tassels, cobs, husks, silks and ears have high percentage of starch and bioactive compounds utilized for the production of feed for humans and animals and to manufacture bio-ethanol. Research studies have been carried out to show the utilization of maize byproducts such as; the growth of effective and consistent removal methods to retrieve phytochemicals and the evaluation of the components of the phytochemicals that is used for the treatment of illness. The health benefits of the phytochemicals include cardiovascular protein effects, anti-aging, anti-apoptosis, anti-atherosclerosis, anti-inflammation and anti-carcinogen which act as antioxidants that are useful to the health of humans (Duangpapeng *et al.*, 2018).

Cellulose, hemicellulose, and lignin are the key components of any lingo-cellulosic resource and the amounts of each of the components on any fiber depend on the time it was plucked (age), source and the process of extraction. Cellulose is one of the major constituents of a maize tassel which is highly renewable, with a linear polymer of b-D-glucose. The production rate of cellulose annually is 1.5 trillion tons from maize tassel, its source is unlimited, ecofriendly and biocompatible (Klemm *et al.*, 2005).

Due to the following qualities maize tassel

process, it has been used by researchers in the research to solve problems. It is a plant based renewable source of adsorbent, it is biodegradable and it has suitable application in reducing hazardous materials. The composition of cellulose, lignin and hemicellulose present in maize tassel fibers are 41%, 18% and 29% respectively (Maepa *et al.*, 2015).

Cellulose surface hydroxyl groups are present in the tassel which is responsible for the bond with positively charged metal ions which implies that it can be useful for bio-sorption process (Zvinowanda *et al.*, 2008; Olorundare *et al.*, 2014).

Bio-sorption is the removal of contaminants from wastes or industrial effluents by living or non-living organisms. These organisms possess various functional groups that attract these contaminants and serve as a great efficiency for their removal (Farooq *et al.*, 2010)

The aim of this study is to investigate the proximate analysis, surface physical and chemistry characterization of maize tassels.

2. METHODOLOGY

2.1 Collection and preparation of maize tassel powder

Maize tassel gotten from a farmland in Chikakore Byazhin, Kubwa Abuja, was placed in a fume cupboard to air dry for two days and rinsed meticulously with 500ml of distilled water. The material was then left to air dry and placed in an oven (Genlab England Model N505F) to dry at 65°C for 5 h to expel any residual moisture. The dried material was then milled by a hammer mill. Thereafter, the milled tassel was sieved to a particle size of 150-500 μm . An electronic weighing balance was used to measure 500g of the milled tassel powder (Sekhula *et al.*, 2012).

2.2 Determination of moisture content

Petri dishes were washed, placed in the oven to dry and left to cool in the desiccator. Each of the dishes was weighed with a digital weighing balance and the mass was recorded and noted as (M_1). An unknown mass of maize tassel powder was placed in the dish and the mass of the dish plus the dried sample was recorded and noted as (M_2). The dried sample containing the maize tassel was placed in an

oven to dry under the temperature of 70°C and it was left over night to dry. The sample was removed the next morning and the new mass was weighed and recorded as (M_3) (Ekpete *et al.*, 2017). The moisture content was calculated as described by equation (1) below:

$$\% \text{ Moisture} = \frac{M_3 - M_1}{M_2 - M_1} \times 100 \quad (1)$$

2.3 Determination of ash content

4g of the maize tassel was placed in a crucible of weight (M_1); then the weight of the crucible plus the sample was weighed and recorded as (M_2). The sample was kindled on a bursen flame inside a fume cupboard to remove some of the smoke, and then it was transferred to a furnace at 650°C for 1 hour until a gray ash colour was noticed. The crucible was placed in a desiccator to cool and the weight was taken and recorded as (M_3) (Ekpete *et al.*, 2017). The Ash of the maize tassel powder was calculated as described by equation (2) below;

$$\% \text{ Ash} = \frac{M_3 - M_1}{M_2 - M_3} \times 100 \quad (2)$$

2.4 Determination of bulk density/ apparent density

An electronic weighing balance was used to weigh 20g of the maize tassel in a 100ml measuring cylinder. The cylinder was tapped 10 times against the palm and placed on the table to record the final volume (Ekpete *et al.*, 2017). The equation below was used to calculate the bulk density;

$$B_D = \frac{\text{Mass of material}}{\text{Volume of material after tapping}} \quad (3)$$

2.5 Determination of Iodine Number

0.5 g of maize tassel powder was transferred to a clean and dry 250-ml Erlenmeyer flask equipped with a ground glass stopper. 10 ml of 5 wt% hydrochloric acid solution was transferred into each of the flask containing the maize tassel. Each of the flasks was stoppered and twirled gently until the maize tassel is completely wetted. The stoppers were undone to vent the flasks, and they were placed on a hot plate in a fume hood and brought to boil. The contents were boiled gently for 30 seconds

to expel any sulfur content which may obstruct the test results. The flasks were removed from the hot plate and left to cool at room temperature. 100 ml of 0.1 N iodine solutions was transferred with the aid of a pipet into each flask and the flasks were stoppered. The contents were shaken briskly for 30 seconds and filtered immediately through a sheet of folded filter paper (Whatman 110mm) into a beaker. 20 ml of the filtrate was used to rinse the pipet and discarded and the remaining were collected in clean beakers. 50ml of the filtrate were transferred into a clean 250-ml Erlenmeyer flask with the aid of a measuring cylinder. Each of the filtrate was titrated with standardized 0.1 N sodium thiosulfate solutions until the solution turns pale yellow. 2ml of the starch indicator solution were added and continue the titration with sodium thiosulfate until one drop produces a colorless solution. The volume of sodium thiosulfate used for determination of the amount of iodine adsorbed on to the carbon (mg/g) was recorded and a blank iodine was titrated with sodium thiosulphate and a starch indicator was used until the solution turns colorless and the volume of sodium thiosulphate used was recorded. The equation below was used to determine the iodine number of maize tassel;

$$\text{Iodine number} = \frac{v \times (T_i - T_f) C_t \times M_i}{T_i \times w} \quad (4)$$

Where v is the volume of iodine, T_i is the sodium thiosulphate solution used for titration of the iodine solution without maize tassel, T_f is the amount of sodium thiosulphate used for titration of iodine and the maize tassel, C_t is the concentration of iodine, M_i is the molar weight of iodine and w is the weight of the maize tassel (Wang *et al.*, 2008).

2.6 Determination of point zero charge

The salt addition approach was done by the inclusion of same quantity of substrate to a set of solutions of similar ionic strength at different pH values. In a series of 50-mL centrifuge tubes, 20.0 mL of 0.1 M NaNO_3 solution was added to a weighed sample of 0.1 g of maize tassel and the pH was altered with 0.1 M HNO_3 and 0.1 M NaOH to achieve the appropriate pH range of 2, 3, 4, 5, 6, 7, 8, 9, 10, and 11 (± 0.1 pH units). The pH values of

the supernatant in each tube were denoted as pH_i . The samples were shaken for 30 minutes using a centrifuge at 3500 rpm. After settling, the pH values of the supernatant in each tube were measured and denoted as pH_f . The PZC was obtained from the plot of ΔpH ($=\text{pH}_f - \text{pH}_i$) against pH_i . Experiments were repeated with a 0.05 M NaNO_3 solution. Each set of experiments was done thrice and the average value was recorded (Bakatula *et al.*, 2017).

2.7 Determination of crude protein

An electronic weighing balance was used to weigh 0.5g of maize tassel and poured into the digestion tube with the addition of 20ml of concentrated Hydrogen tetraoxosulphate (VI) acid. A selenium tablet was added to the mixture as a catalyst. The content in the tube was heated at a temperature of 350°C for 6 hours until a clear solution (digest) was achieved. This solution was poured into a measuring cylinder and made up to 100ml. 10ml of 2% boric acid was taken into a 100ml conical flask and added with three drops of mixed indicator (Bromocresol green and Methyl red) and the color changes to pink which was then placed under the collecting spot. 10ml of the digested sample was pipetted into the open chamber of the makhamps apparatus then followed by 10ml of 40% NaOH . The mixture was hereby boiled by the steam produced by the boiling water in the flat bottom flask. As the mixtures boils, a gas (ammonia) was evolve and condense by the condenser of the apparatus which was collected in form of liquid into the boric acid. As the ammonia is collected in the boric acid, the solution turns blue. The distillate collected was titrated using 0.1M HCl until an end point is reached by the colour change of the distillate to pink colour. Crude protein is calculated with the equation below;

$$Cr = \frac{TV \times 0.014 \times M_A \times D_F \times 100}{S}$$

Where TV is the titre value of hydrochloric acid used, 0.014 is nitrogen standard, M_A is the molar concentration of hydrochloric acid, D_F is the dilution factor and S is the weight of maize tassel used (Onwuka, 2005).

2.8 Determination of oxygen containing functional groups

The Boehm titration method was used for this analysis. 1.0g of the maize tassel were added to each 15ml solution of NaHCO_3 (0.1M), Na_2CO_3 (0.05M) and NaOH (0.1M) for acidic groups and 0.1M HCl for basic groups/sites respectively at ambient temperature for more than 2 days. Direct titration process was carried out to determine the functional groups present. The number and type of acidic sites were calculated in view that NaOH neutralises carboxylic, lactonic and phenolic groups, Na_2CO_3 neutralises carboxylic and lactonic groups and that NaHCO_3 neutralises only carboxylic groups. Carboxylic groups were therefore quantified by direct titration with NaHCO_3 . The change between the groups titrated with Na_2CO_3 and those titrated with NaHCO_3 was inferred to be lactones and the change between the groups titrated with NaOH and those titrated with Na_2CO_3 was presumed to be phenol. Neutralisation points were known using pH indicators of phenolphthalein solution for titration of strong base and strong acid, methyl red solution for weak base with strong acid and pH together. In order to neutralize basic groups sites, remaining HCl in the solution was back titrated with 0.1M NaOH (Ekpete and Horsfall, 2011).

The equation below was used to solve for the functional groups present in the maize tassel;

$$n_{\text{csf}} = [B]V_B - [\text{HCl}]V_{\text{HCl}} \frac{V_B}{V_A} \quad (6)$$

Where $[B]$ is the concentration of base, V_B is the volume of the reaction base, n_{csf} is the moles of maize tassel surface functionalities on the surface of carbon that reacted with the base, V_a volume of the aliquot taken from V_B , $[\text{HCl}]$ is the concentration of HCl and V_{HCl} is the volume of acid used (Goertzen *et al.*, 2009).

2.9 Determination of surface functional groups

FT-IR spectra were used to study the structure of maize tassel fibers using a Nicolet 560 spectrophotometer. The size of the maize tassel fibers were reduced and mixed with KBr powders and the mixture was compressed into plates for FT-IR analysis. The FT-IR spectra of the samples were obtained in the wavelength range of 4000–650 cm^{-1}

1. A total of 32 scans were co-added in order to achieve an acceptable signal-to-noise ratio. In all cases, spectra resolution was maintained at 4 cm^{-1} (Mwangi *et al.*, 2012).

2.10 Determination of surface area and pore volume

The porosity and specific surface area were determined by the modeling of N_2 -adsorption data with the Brunauer–Emmett–Teller (BET) isotherm. The particle size distribution was gotten by the fitting of an algorithm to patterns generated by the diffraction of a laser beam by suspended adsorbent particles. A Novastation D (version 11.03) was used to achieve textural analysis on the maize tassel powder determined by nitrogen adsorption–desorption at an analysis bath temperature of 273k, outgas temperature of 250 °C, analysis time of 79 minutes and an equilibration interval of 12 s. The sample was automatically degassed. A sample mass of 0.08 g was used and the adsorptive properties of nitrogen were analyzed. The multiple-point BET surface area, adsorption total pore volume and the pore diameter were determined (Zvinowanda *et al.*, 2008).

3.0 Results and Discussion

3.1 Proximate analysis of maize tassel

Adsorbents materials are generally valued on a moisture free basis but some moisture content is stipulated except its being packaged in air tight containers. They can adsorb moisture and still remain dry, this moisture content doesn't affect the adsorptive power but it reduces the carbon content present in the adsorbent materials. The permissible limit of moisture content allowed in adsorbent materials is within the range of 3-6% (ISI standards).

This statement is true when taking maize tassel into consideration. The values of the surface characterization of maize tassel are shown in table 3.1 below;

TABLE 1 : SURFACE CHARACTERIZATION OF MAIZE TASSEL

Parameter	Value
Moisture content (%)	4.51
Ash content (%)	2.134
Apparent density (g/cm ³)	0.41
Iodine number	423.049
Particle size (µm)	300
Crude protein (%)	4.06
Acid soluble matter (%)	79
Water soluble matter (%)	93.7

From the table above, it shows that maize tassel moisture and ash content are at a low level having values of 4.51% and 2.134% respectively.

According to Ekpete *et al.*, (2017) the presence of low amount of moisture and ash content present in adsorbent materials shows that the density of the particles are small and it would be a remarkable unprocessed material for adsorption purposes in a fixed bed or column reactor. The lower the moisture and ash value the greater the adsorbents and the value should be within the ranges of 1-20 % hereby making maize tassel a viable sorbent for the extraction of pollutants from waste water.

The apparent density is a vital physical parameter which evaluates the filterability of a sorbent material because the amount of carbon present in a filter of given solids can be determined by the amount of treated liquid retained by the filter cake. The apparent density of the adsorbent material has an effect on adsorption per unit volume but does not affect the efficiency of measured in adsorption per unit weight. If the density of the material is high, small amount of the sample is sufficient for carrying out adsorption studies (Ekpete and Horsfall, 2011).

From table 1 above the apparent density of maize tassel is 0.41g/ml.

The acid soluble matter is vital because when the

waste water being treated is acidic, the adsorbent becomes soluble and the effluent contains the material. Maize tassel cannot be used efficiently adsorption in an acidic medium because it is highly soluble in acid.

The water-soluble matter of the maize tassel was determined and the results were given in the table 1 above. This is an essential property to be considered because when it is used as an adsorbent for water treatment, it should not be soluble in water. From the results gotten in table 1 above, maize tassel is highly soluble but to be used in water treatment it has to be modified any sample must be least soluble in water. It was reported by Agyei *et al.*, (2016) that activated maize tassel removed phosphates at a faster rate and with higher efficiency than raw maize tassel.

3.2 Surface characterization of maize tassel

Iodine number is used solely for the purpose of evaluating the surface area of sorbent materials at ambient temperature conditions to aid in evaluating the porosity and adsorbent capacity of the materials. The increase in iodine number of carbon atoms has been attributed to the occurrence of enormous micro-pores structure and to the immense possibility of carbons present in the adsorbent materials to have enormous surface area as a result of the increase of their pore structure (Ekpete *et al.*, 2017).

As observed in the table above, the iodine number for maize tassel is 423.049 which shows that it has large micro-pores thereby leading to large surface area which is a necessity for adsorption process.

The capability of an adsorbent material to adsorb contaminants is an essential property due to its specific surface area. Mostly, the increase in surface area of the sorbent material determines the capability of adsorbing more contaminants. The table 3 below shows the result of the surface area and pore volume on the maize tassel;

TABLE 2: ADSORPTION PARAMETERS OBTAINED BY THE APPLICATION OF THE BET MODEL TO ADSORPTION ISOTHERMS OF MAIZE TASSEL

Sample	Surface area (m ² /g)	Pore volume (cm ³ /g)	Pore size (nm)
Maize tassel	652.3	0.4056	2.144

The point of zero charge is an essential measurable property of any sorbent material. They are pH values at which the surface charge components sum up to 0 under given conditions such as temperature, pressure and composition which implies equal amounts of positive and negative charges. At pH below the point zero charge the sorbent material is positively charged which implies the material will adsorb anions while at pH values above the point zero charge the sorbent material is negatively charged which results to the material adsorbing cations.

The point zero charge values might aid in the selection of a material for the removal of pollutants from effluents. Sorbent materials that have low point zero charge would be appropriate to remove pollutants that possess positive ions, while materials with high PZC values would be best to remove negative ions (Bakatula *et al.*, 2017).

The curves gotten from the (plots of ΔpH vs pH_i) following the salt addition technique at both ionic strengths of (0.05 and 0.1M of NaNO₃) were done in triplicates and the average was taken are presented in figure 3.1 below;

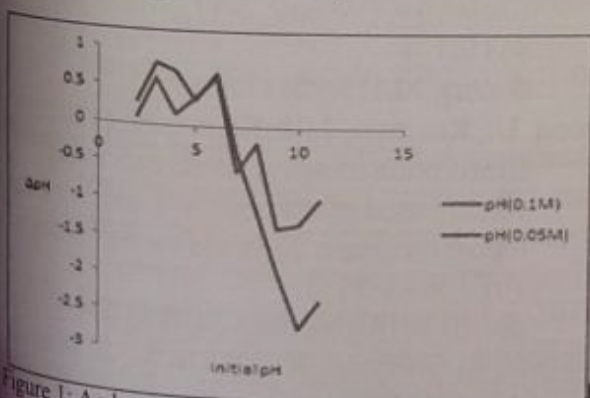


Figure 1: A plot of the Change in Final pH and Initial pH against the Initial pH for maize tassel

In 0.05 M NaNO₃, the point zero charge values of maize tassel was 6.8. When the ionic strength was increased to 0.1 M, the point zero charge reduced to 6.6 with a minor decrease of PZC (0.2 pH unit).

According to Sillero *et al.*, (2013) the point zero charge of peat and sawdust using the same ionic strength and same salts as stated above were 3.42 & 4.26 respectively.

Studies carried out by Cristiano *et al.*, (2011) yielded point zero charge of 7.2 using the salt addition method with ionic strength of 0.01–0.001 M NaClO₄.

3.2 Surface chemistry characterization

Boehm's method gives a semi-quantitative degree of surface functionalities since the chemical groups are more difficult and are shown in Boehm titrations. With NaHCO₃, Na₂CO₃, NaOH, and HCl assumed to be neutralizing carboxylic groups, lactonic and carboxylic groups, lactonic, carboxylic, and phenolic groups, and all basic groups, the nominal observation of the values showed that there was no much change existing between the carbon samples when the acidic sites are taken into consideration. The table 3 below shows the oxygen functional groups of maize tassel plant below;

TABLE 3: THE OXYGEN FUNCTIONAL GROUPS OF MAIZE TASSEL

Groups	Maize Tassel (mmol/g)
Carboxyl	0.9
Lactones	0.6
Phenols	-0.66
Total non-carbonyl	0.84

The oxygen containing groups were analyzed and evaluated with the aid of FT-IR. Due to the distinctive property of absorbing energy from the different bonds in each group, FTIR spectra discloses the specific surface functional groups on the maize tassel fibre (Ismadji *et al.*, 2010)

The affirmations of the chemical structures of maize tassel fibers were gotten from FT-IR analysis. The composition changes observed for maize tassel fibers are shown in figure 2 below;

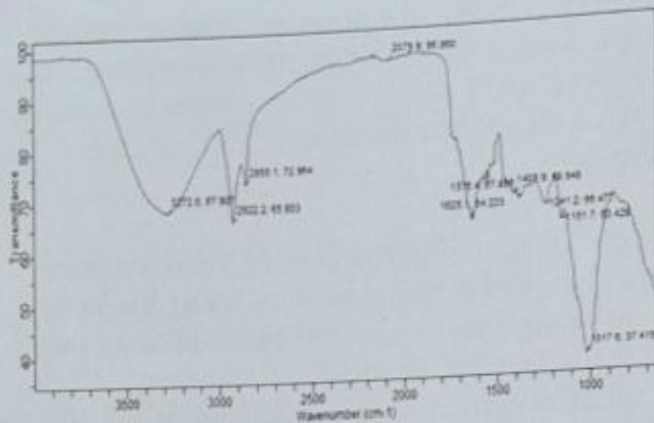


Figure 2: FT-IR spectrum of maize tassel

TABLE 4:
THE BONDS PRESENT IN MAIZE TASSEL FIBRE

Transmittance	Maize tassel peaks (cm ⁻¹)	Bonds indicative
67.907	3272	Carboxylic and alcohol stretching vibration
65.803	2922.2	Alkanes stretching vibration
72.964	2855.1	Alkanes stretching vibration
64.223	1625.1	Alkenes (CH ₂) bending vibrations
66.477- 63.428	1241.2-1151.7	Alkyl, Aryl or Ether stretching vibration

The spectrum shows the presence of hydroxyl, carbonyl, ether groups, and absorbed water in the maize tassel fiber which can be bonded with cations easily (Zvinowanda *et al.*, 2008).

As shown in the Figure 2 above, the hemicellulose intensity of the peak appeared at around 1730 and 1240cm⁻¹ in the maize tassel which is in agreement to Obi *et al.*, (2014).

The functional groups present in maize tassel fiber presented in figure 2 above indicates strong bonds between 3272 and 2922 cm⁻¹ attributed to carboxylic groups, alcohol, alkanes or Amine groups (I.R Spectrum table and chart, n.d).

The two bands at 2855.1 and 1625.1 cm⁻¹ were ascribed to the alkanes and alkenes groups respectively while the band at 1625.1 cm⁻¹ which was assigned to stretching of carbonyl group. This work is in agreement with the reports of Mwangi *et al.*, (2012).

4.0 Conclusion

Adsorption is reliant on material characteristics such as surface area, particle size and number of carbon atoms present. In this study, characterization was carried out on maize tassel fibre and the results of the BET and Iodine number indicate that maize tassel fibre is mesoporous and a good material for bio-sorption process. The results from this study have shown the prospects of maize tassel to be used as a low-cost and effective bio-sorbent for the removal of contaminants. Further studies on the application of the material will reveal the extent of removal based on the adsorbent dosage and concentration of the sorbate. Thus, the permissible limits from environmental wastewaters can be identified. The effectiveness of the adsorbent can enhance the safety of industrial extent before the discharge of industrial effluents into water bodies.

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