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Surface Properties and Chemical Constituents of Unmodified and Oxalic Acid Modified Cassava (*Manihot esculenta crantz*) Peel Waste Powder

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Abstract: Surface chemistry plays a vital role in various industrial technologies such as chemical and energy conversion, health care, and material and environmental protection. This research investigated the surface properties and chemical constituents of unmodified and oxalic acid modified cassava peel waste powder (CPP) using standard methods. Results show that the surface chemical content were ash content: ash (0.82%), moisture (12.39%), sodium capacity (0.13 mMole/g), apparent density (1.29 g/cm^3) and pore volume (0.78 cm^3/g) for unmodified CPP while for modified CPP, the contents were Ash (0.55%), moisture (17.46%), sodium capacity (0.10 mMole/g), apparent density (1.20 g/cm^3), and pore volume (0.83 cm^3/g) respectively. FTIR analysis reveals the major bands and their significance and showed existence of ionizable functional groups as follows: -OH was observed in the range of 3000-3700cm^{-1}, from 600-1400cm^{-1} in the fingerprint range C=C, C=O and C=N bonds were suspected. All functional groups can take part in adsorption process through ion-exchange mechanism or complexation mechanism The CPP biomass had intense bands at 2924cm^{-1} indicating presence of amines (N-H) functional groups. The surface chemistry further revealed that cassava peel powder is an excellent potential biomaterial for diverse purposes.

Keywords: Surface Chemistry, *Manihot esculenta crantz*, Cassava Peel Wastes

1. Introduction

Surface properties and chemical constituents are crucial parameters for assessing biomaterials and their adoption as industrial precursors. Horsfall and Spiff [1-9]; Horsfall et al [10-13] investigated the use of many agricultural wastes as adsorbents for the removal of inorganic (heavy metals) and organic (dyes) contaminants from wastewater. Other agricultural solid wastes from cheap and readily available resources such as agave bagasse [13-15], almond shell [16], apricot shell [16], barley straw [17], cashew nut shell [18], citric acid [19-20], corncob [21], garden grass [22], mango peel waste [23], muskmelon peel [24], pine sawdust [25], groundnut shell [15], plum kernel [13], potato peel [27], rice straw [28], sugarcane bagasse [29, 30], banana peel [31], orange peel [32, 33, 34, 35, 36, 37], and coconut shells [38] have also been investigated for the removal of numerous heavy metals and dyes from aqueous solutions. The basic components of the agricultural waste materials include hemicelluloses, lignin, lipids, proteins, simple sugars, water, hydrocarbons and starch, containing a variety of functional groups with a potential sorption capacity for various pollutants [39, 40]. Agricultural waste products are used in the natural and modified form. In the natural form, the product is washed, ground and sieved until reaches the desired particle size and subsequently used in adsorption tests. While, in the modified form, the product is pre-treated by-means of well-known modification techniques [40]. The goal of these pretreatments is to enhance and reinforce the functional group potential and, consequently, increase the number of active sites. Agricultural waste products have been extensively studied in relation to the adsorption process.

Cassava (Manihot esculenta, Crantz) is an important food security crop, but it is becoming an important raw material for different industrial applications. Cassava is the second most important source of starch worldwide [41]. According to Haggblade et al [42], cassava (Manihot esculenta, Crantz) is an important source of food calories in sub-Saharan Africa, fulfilling a critical role as a food security crop.

Industrial and traditional processing of cassava into its various products produce huge wastes, which creates a lot conservational problem. So far, not much effort is made to manage the enormous waste arising from cassava processing.

Hence, the objective of this study is to characterize the Surface properties and chemical constituents of unmodified and oxalic acid modified cassava (*Manihot esculenta crantz)* peel powder with a view of creating a better sustainable adsorbent for waste water treatment process.

2. Materials and Method

Sample Collection: The cassava tubers used for this project work were gotten from traders in Choba market near University of Port Harcourt in Rivers State and identified in the department of plant science and Biotechnology University of Port Harcourt.

Sample Preparation: The obtained cassava was cut into small pieces, washed to get rid of sand and dirt particles and were peeled to get the powders. The air-dried cassava peel powder was further dried in oven (Gallenkamp, model OV-160 England) at 105°C to a particular weight and pulverized with a grinder to get a standardized particle size.

Preparation of Cassava Peel Powder: The ground cassava peel powder (CPP) was soaked in 0.1M HNO_3 acid for 48 hours so that all metals contained in the CPP can be removed, followed by a thorough wash with distilled water. The washing continued until the filtrate gave a negative EDTA test for heavy metal ions, by adding 5 drops of 0.01M EDTA solution and 2ml of NH_4Cl buffer to 5ml of the filtrate. Blue color of EDTA solution showed no presence of metal ions. The Cassava peel paste was dried at 45°C in oven, and meshed. The portion with size <100µm was taken for use and was kept in clean air-tight plastic containers for use.

Preparation of Modified Cassava Peel Powder: 10 ± 0.12 grams of the cassava peel powder (CPP) was weighed into 250ml beakers and was modified with 250ml of 0.5 M of oxalic acid solution respectively with one portion left unmodified. The mixture was shake for six hours at 30°C and was kept stable overnight, discarding the filtrate and the Cassava Peel paste was air- dried to obtain the modified cassava peel powder (CPP).

Characterization of Unmodified and Modified Cassava Peel Powder: The different modified CPP were characterized to determine the physicochemical properties.

Determination of Moisture Content: Moisture content was determined by oven- drying technique using two grams of CPP in dry crucible (W_1) at 105°C for six hours, it was cooled and weighed as (W_2). The moisture content was evaluated using the equation:

$$\%\ \text{Moisture} = \frac{W_1 - W_2}{\text{wt.of sample}}\ x\ 100 \qquad (1)$$

Where: W_1, Initial weight of crucible and Sample; W_2, Final weight of crucible and Sample.

Determination of Ash Content: For ash content analysis, empty crucible was put in a muffle furnace at 600°C for one hour, cooled, weighed, and recorded as (W_1). Two grams of CPP was taken into the crucible, and then placed in furnace at 550°C for three hours. Gray color of the sample specifies total oxidation of all organic material in the sample, this was cooled and weighed (W_2), and calculated using:

$$\%\ \text{Ash} = \frac{\text{Difference in wt of Ash}}{\text{wt of sample}}\ x\ 100 \qquad (2)$$

Difference in weight of Ash= $W_2 - W_1$

Determination of Particle Density: The unmodified and different modified cassava peel powder of known weights (Wa). Two grams of CPP was put into a measuring cylinder, and tapped gently and volume of the CPP was recorded as V_a. A known volume of purified water was added slowly through the side of the cylinder to soak the sample properly. The final volume containing CPP was recorded as V_b. The particle density is calculated as:

$$P\ (g/cm^3) = \frac{Wa}{Vb - Va} \qquad (3)$$

Determination of Sodium Capacity: This was evaluated using ten grams of the cassava peel powder with twenty-five milliliter of 0.1M NaOH. The solution was stirred on a shaker for Seventy-two hours, and then filtered to remove the adsorbent, twenty milliliters of the filtrate was taken and titrated with 0.1M HCl and methyl orange as an indicator. The sodium content was calculated appropriately [43].

Determination of Surface Functional Groups on the Unmodified and Modified CPP using FT-IR: Fourier transform infrared spectrometer (Tensor 27 Bruker) in the range of 4000–400cm^{-1} was used for the evaluation of surface functional groups of unmodified and modified Cassava Peel powder. The samples were prepared as pellets using KBr (IR grade).

Statistical Evaluation of Experimental Data: Mean, Standard deviation and Analysis of Variance (ANOVA) was employed in the evaluation of experimental data.

3. Results and Discussion

Physicochemical Properties of Unmodified and Modified Cassava Peel Powder: The data in table 1 show some physicochemical characteristics of the cassava peel. These properties are vital in adsorption of metal ions unto the CPP.

Table 1. *Physiochemical properties of unmodified and differentially modified CPP.*

Adsorbents	Ash (%)	Moisture (%)	Sodium Capacity mMole/g	Apparent density g/cm^3	Pore Volume cm^3/g
Unmodified CPP	0.82	12.39	0.13	1.29	0.78
0.5M Modified CPP	0.55	17.46	0.10	1.20	0.83

The results of the ash content percentage (which is the inorganic residue remaining after the organic matter has been burnt off) [44, 45]. was determined and found to be; 0.82% for the unmodified cassava peel powder and 0.55% for 0.5M Oxalic acid modified cassava peel powder. The data indicates that increase in concentration of modifying oxalic acid led to decrease in ash of biomass. The low ash value shows that CPP can be easily suspended in aqueous system. A reduction in ash content improves adsorption on the adsorbent. [46] (Lin and Wu 2001).

Moisture content in a sample refers to amount of water in the sample. The observed rise in moisture content of modified CPP was because of increase in concentration of the oxalic acid. Increase in concentration of oxalic acid results in increase in moisture level of CPP, this indicates that CPP can be suspended in liquid phase. For enhanced adsorption capacity, content of moisture in adsorbents must be relatively low [43]. The high moisture content can be attributed to the fact that the sample was collected in rainy season when the rate of evaporation is very low as a result of high atmospheric humidity.

The CPP showed affinity for sodium ions (Na^+). This is analyzed to assess the total sorption ability of CPP for cations, the sodium uptake is because of weakly acidic surface functional groups on the CPP, which when in solution may easily dissociate and become ion exchange spots for metal ions. Based on result, the lower the concentration of modified CPP, the higher the sodium capacity. This further indicates greater sorption capacity of CPP [43].

Density is a significant physical parameter especially when an adsorbent is being considered for its filterability [47]. High density and pore volume provide greater volume activity and normally indicates high quality adsorbent. The obtained results for the cassava peel in the range of 1.20 - 1.33 g/cm^3 were favorable because it revealed that it was good adsorbent in terms of volume activity, since adsorption is a surface phenomenon [48]. These results are in agreement with Shakirullah and co-wokers [50] of 1.26 g/cm^3 for sawdust of *Dalbergiasissoo* and Abechi [49] who reported 1.1034 g/cm^3 for walnut shell. Horsfall and Vicente [43] also reported 1.2 g/cm^3 for almond tree leaf. Furthermore, Horsfall and Vincente [43] reported that particle density close to unity indicates high interaction between the adsorbate and adsorbent. The pore volume is an inverse relation of particle density. The pore volume is vital in determining the pore arrangement of biomass [50].

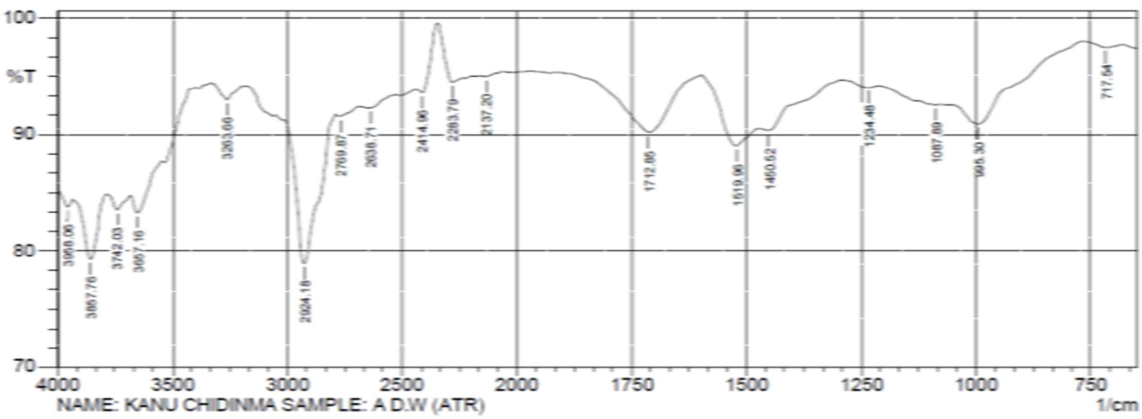

Figure 1. *FT-IR spectra for unmodified CPP.*

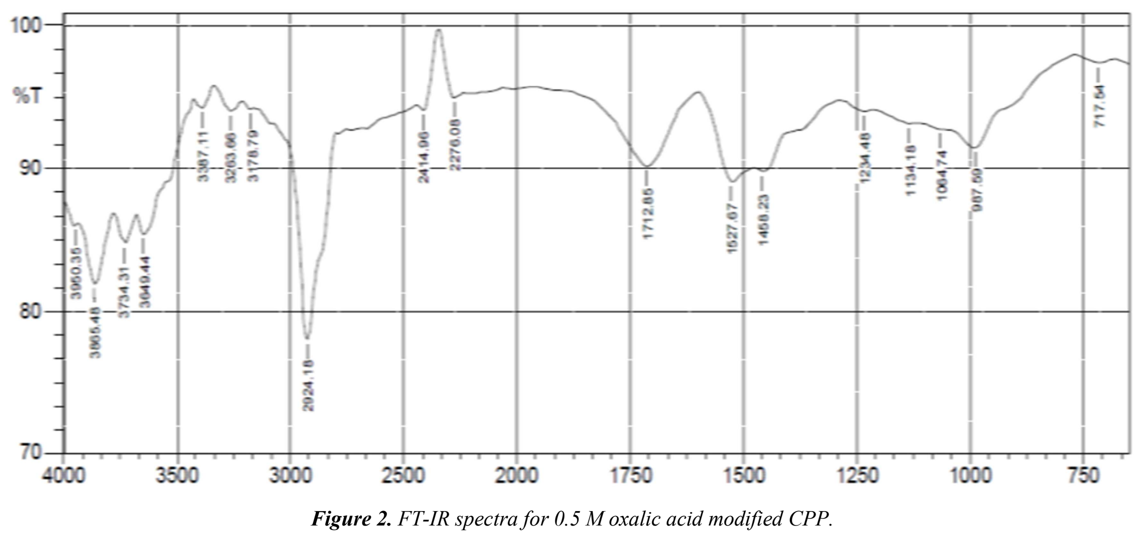

Figure 2. *FT-IR spectra for 0.5 M oxalic acid modified CPP.*

Modification with oxalic acid reduced the sorption of prepared CPP because of protonation of sorption sites of CPP surface due to modification with acid. This will tend to leave metal ions in solution rather than being adsorbed on the surface [51]. Rate of metal sorption on CPP from solution depends on the nature of the metal. This relates to the ability to make complexes with functional groups on CPP surface [52].

Surface Chemical Composition of Unmodified and Modified Cassava Peel Powder: The surface functional groups of the unmodified and differentially oxalic acid modified cassava peel powder were evaluated by Fourier Transform Infrared (FTIR) Spectroscopy. FTIR is an effective tool to determine the surface chemical composition of rigid materials. The analysis by FTIR constitutes the absorbance of the sample to an incident infrared spectrum in the range of 400 to 4000cm^{-1} The FTIR spectra for unmodified and oxalic acid modified cassava peel powder are presented in Figures 1 and 2 respectively. The FTIR analysis was performed on unmodified and oxalic acid modified CPP to determine the functional groups inherent in them. This was done to study the impact and effect that the modification process had on the original material (cassava peel). The FTIR spectra of the two materials are shown in figures 1 and 2 reveals that the FTIR spectra of unmodified CPP spectra at characteristic peaks of 3250.2 cm^{-1} and 2929.7 cm^{-1} showed the presence of hydroxyl groups, while the characteristic peak at 2109.7 cm^{-1} corresponds to the C=N stretching vibration, observed in inorganic cyanide. The characteristic peak at 1636.3 cm^{-1} speaks of the presence of the alkene functional group. While the peak at 1420.1 cm^{-1} was assigned to C=O in-plane bending and 1338.1 cm^{-1} was assigned to the stretching vibration of S=O, the peaks at 1148 cm^{-1} and 1077.2 cm^{-1} were assigned to C=O in-plane stretching. These oxygenated groups represent the presence of the lignin faction of the cassava peel. The bands at 861 cm^{-1}; 764.1 cm^{-1}, and 704.5 cm^{-1} correspond to the presence of alkene hydrogen in out-of-plane bending mode.

The FTIR spectra of oxalic acid modified CPP showed that the modification process introduced new peaks into the biomass observed at 3801.9 cm^{-1}, 3548.4 cm^{-1}, 3116.1 cm^{-1}, and 1230 cm^{-1}. The loss of some peaks in CP, such as 704.5 cm^{-1}, 928.1 cm^{-1}, and 1077.2 cm^{-1}, was also observed. Shifts in peaks were also observed from 3250.2 cm^{-1} to 3343.4 cm^{-1}, 2929.7 cm^{-1} to 2918.5 cm^{-1}, 2109.7 cm^{-1} to 2102.2 cm^{-1}, 1636.3 cm^{-1} to 1558 cm^{-1}, 1338.1 cm^{-1} to 1375.4 cm^{-1}, 861 cm^{-1} to 872.2 cm^{-1}, and 764.1 cm^{-1} to 752.9 cm^{-1}, for unmodified CPP and oxalic acid modified CPP, respectively.

4. Conclusions

The physiochemical evaluation of the cassava peel showed that the CPP is a potential material as adsorbent based on its volume activity and suspension in liquid phase. The major bands and their significance showed existence of ionizable functional groups that could interact with metal ions in an adsorption process. In all-purpose, it was observed that Unmodified CPP and Oxalic acid modified CPP have similar functional groups, however, with more of the peaks representing oxygenated groups in oxalic acid modified CPP than those in unmodified CPP.

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