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SYNTHESIS AND CHARACTERIZATION OF A HIGH SURFACE AREA ACTIVATED CARBON DERIVED FROM CASSAVA PEELS WASTE BY KOH ACTIVATION

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ABSTRACT

This work presents the synthesis of activated carbon, a very important material with lots of use cases in textile, food industries and even in energy storage as an electrode material in supercapacitor development. Abundantly available cassava peels was used as a raw material for the production of the activated carbon by adopting the chemical activation approach which utilized KOH as activating agent. The activation was carried out at 800^0C in a tubular furnace. Scanning electron micrographs (SEM) showed that a highly porous carbon structure was achieved with high specific surface area of $828m^2g^{-1}$ as evident from the BET results. Raman spectrophotometer analysis showed that the prepared activated carbon was highly graphitized as seen from the G peak. The existence of mesopores and micropores was confirmed by the N_2 adsorption/desorption analysis which also revealed the presence of a hysteris loop and a $P/P_0=0.45$ value.

Keywords: [Cassava peels, Chemical Activation, Activated carbon, Porous carbon]

INTRODUCTION

Activated carbons are widely explored in many fields due to their inherent properties which are good porosity and surface area. These properties make them serve as excellent adsorbents in the removal of pollutants and purification in both gaseous and liquid media (Nwabanne and Igbokwe, 2011). Amongst other industries, the textile, electroplating and food industries regularly discharge colored waste water and effluents which if left untreated pose life threatening risks to the environment due to their toxicity.

Treatment by biological means, coagulation, oxidation, membrane filtration and adsorption are some of the most commonly used techniques for these effluents. Of all these methods adsorption is most adopted perhaps due to its ability to remove even color from the pollutant and activated carbon is arguably the most widely used for its adsorbent properties (Sevilla and Fuertes, 2014).

Another growing field of application of activated carbon is the renewable energy field, where lack of proper energy storage systems have continually hampered the switch from commonly used energy systems (fossils) to their renewable counterpart. With the advent of electric vehicles and electronic devices becoming more portable, efficient energy storage devices have become very important. Batteries have long played this role but for their poor power densities, reliance is now shifted to the Electrochemical Double Layer

capacitors (EDLC) otherwise known as supercapacitors due to their high power densities and long cyclability (Yao et al., 2016).

EDLC electrodes can be easily made from activated carbon and this has opened up another broad research area to not only make the activated carbon produced for this purpose environmentally friendly, but also very cheap and readily available.(Sevilla and Mokaya, 2014) The catch in this interest is the porosity, good electrical conductivity and electrical stability of activated carbon and the high carbon content which is suitable for energy storage (Moyo et al., 2017).

Preparation of commercially available activated carbons actually go through a whole lot of complicated procedures which pose the risk of keeping the developing countries behind in application (Ismanto et al., 2010). For this reason, an easy approach and abundance based method is suggested. Using easily assessable biomass as starting materials will prove cost effective and show availability at all times. So many biomass has been reportedly used in the preparation of activated carbon. Materials such as bamboo (Ma et al., 2014), olive stones (Yakout and Sharaf, 2016), nipal palm nut (Nwabanne and Igbokwe ,2011), corncob (Yang and Zhang, 2018), rice husk (Huang et al., 2017) and hydrochar (Sevilla and Fuertes, 2014.) have all been used to produce activated carbon, using the two main methods of activation reported by Sevilla and Mokaya (2018).

There are two main approaches to preparation of activated carbon; chemical activation and physical activation. Physical Activation utilizes a process whereby carbonaceous material is carbonized which is followed by an activating process in the presence of steam or CO_2. This is done at elevated temperatures where the precursor is made to have contact with the gas or steam for activation (Ponkarthikeyan and Sree, 2017). In physical activation, the processes of carbonization and activation are two separate processes. In Chemical activation, the precursor is impregnated with activating agent ($ZnCl_2$, H_3PO_4, KOH) which then undergoes heat-treatment in an inert environment. In this method, the activation and carbonization occur simultaneously and at lower temperatures when compared to the physical activation process, leading to better pore structure development in the carbon material (Lamine et al., 2014). Physical activation produces carbons with porous structures made up of mostly micropores (<2nm), while chemical activation produces more versatile materials having larger surface area, with porosities made up of mesopores and micropores.

In this study, cassava peels is used as precursor in the production of activated carbon by chemical activation using KOH as activating agent as seen in Scheme 1. Cassava is by far the most abundant agricultural produce in Nigeria which appears in various forms of food in every household across the country. However, its peels pose grave challenge to the environment as an agricultural waste and disposal is always a challenge. Recycling these wastes to a useful venture will be of high economic and research importance to the nation.

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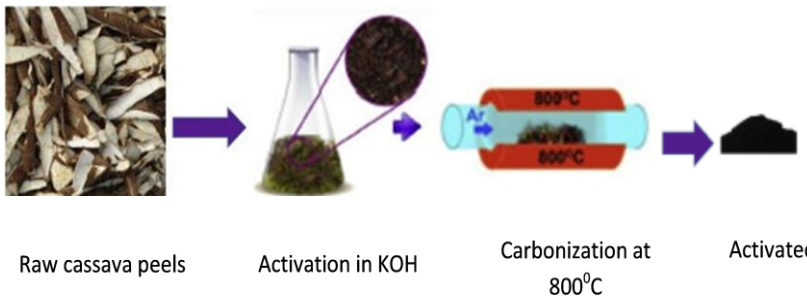

Scheme 1: production of activated carbon from cassava peels

MATERIALS AND METHODS

Preparation and Synthesis of Activated Carbon from Cassava Peels

Freshly uprooted cassavas were collected from local farmers in the sub-urban Abuja, Nigeria. The peels were continuously washed until all forms of dirt were removed and subsequently oven dried for 12 hours at 60^0C.The dried samples were then first crushed with a jaw crusher and further ball-milled to achieve fine powder form. Approximately 3g of the carbon precursor (cassava peels) was measured and poured into a mortar together with 12g of KOH pellets (1:4 ratio) and mixed to form a uniform powder. The mixture was then carbonized under Nitrogen gas flow in a Carbolite tube furnace at 800^0C at a 5^0Cmin^{-1} heating rate and left to carbonize for one hour. The activated carbon was then removed from the furnace tube, allowed to cool to room temperature and washed with 1M HCl to remove any inorganic salts and then washed with distilled water continuously until a Neutral pH was achieved. The sample was finally oven-dried for 24 hours at 80^0C.

Proximate analysis

Proximate analysis of cassava peel showed that the moisture content, fixed carbon, ash content, and the volatile matter were 4.5%, 80.53%, 5.28% and 9.62% respectively as represented in table 1.

Table 1: Proximate analysis of sample.

Moisture content	Fixed carbon	Ash content	Volatile matter
4.57%	80.53%	5.28%	9.62%

Physico-chemical Analysis

An X-Pert Pro instrument was used to carry out X-ray diffraction (XRD) studies of the activated carbon samples, with a reflection geometry of 2θ values (10-80^0) and a radiation (Cu- Kα) source having a 0.15418nm wavelength (λ). Also, using a Jobin Yvon Horiba TX 6400 Raman spectrophotometer, Raman spectroscopy analysis was carried out at a 15mW power rating to avoid overheating of the sample. A Zeiss Model EVO LS10 Scanning Electron Microscope was used for SEM analysis of the prepared materials to get further insight on the structure of the samples. Adsorption properties of the prepared activated carbon material were studied by adopting Nitrogen adsorption/desorption techniques using a NOVATOUCH gas adsorption analyzer. To achieve this, the samples were first degassed for 6hrs in a high vacuum

environment at 100^0C. Furthermore, the specific surface area of the samples was evaluated using the globally adopted BET (Brunauer-Emmett-Teller) method and the pore size distribution was subsequently determined by adopting the BJH (Barrett Joyner-Halenda) method.

RESULTS AND DISCUSSION

Table 2: EDS elemental composition of carbon activated with KOH

Sample	C	Na	Si	Mo	S	Ca
KOH Activated	87.19	-	0.5	7.61	0.01	4.68

Table 3: BET surface area and porous textual data obtained for cassava peel derived carbon material activated at 800^0C.

Sample	KOH activated cassava peels
BET (m^2g^{-1})	828
Micropore volume (cm^3g^{-1})	0.3981
Pore diameter (nm)	2.66

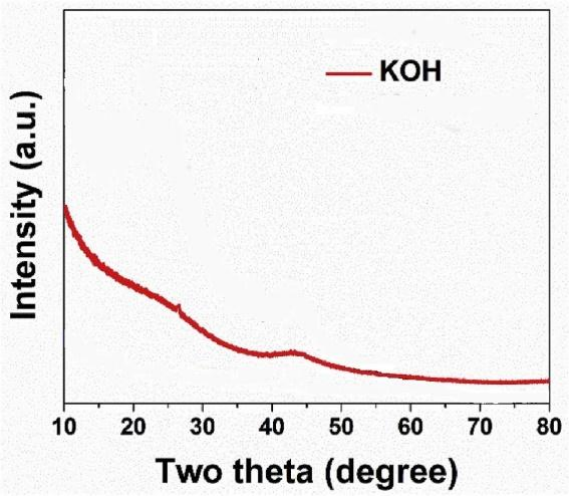

Figure 1: XRD analysis of activated carbon samples

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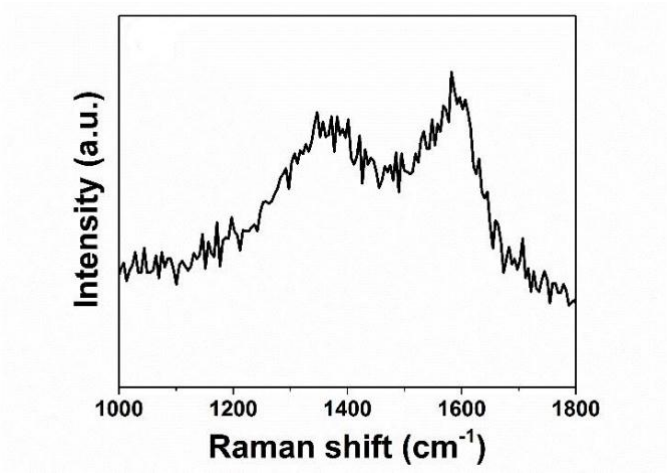

Figure 2: Raman spectra of the sample

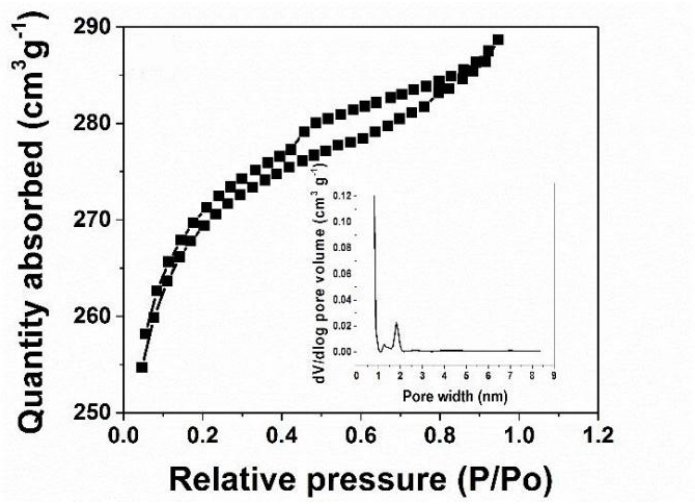

Figure 3: N_2 adsorption/desorption analysis

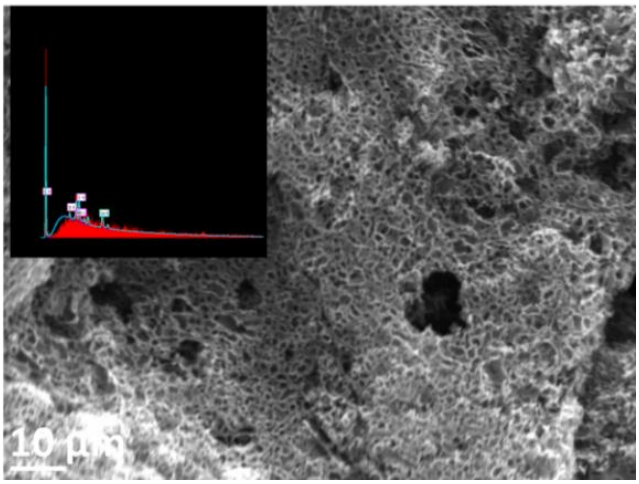

Figure 4: SEM analysis of the samples with inset of EDS

The ash content, moisture content, volatile matter and fixed carbon contents of the cassava peels was ascertained by carrying out proximate analysis using the ASTM methods (Kumar and Jena, 2016). The fixed carbon content of the sample was calculated by mass difference. The proximate composition of the material is as presented

in Table 1. The suitability of cassava peel as precursor for activated carbon was confirmed by these results which showed that the fixed carbon content in the raw material is 80.53wt %.

XRD analysis was carried out to confirm the phase composition of the material. As can be seen from Figure 1, the XRD patterns for the prepared AC sample showed an amorphous signature (i.e. broad diffraction peaks) at a 2-theta angle of 24–25^0 and 42–44^0 corresponding to the (002) and (100) crystallographic planes of a carbon material (Barzegar et al., 2016). The degree of graphitization of the carbon material was confirmed by using Raman spectroscopy as shown in Figure 2. The Raman shift as displayed shows the conspicuous peaks representing the G (graphitic) and D (diamond) phases of carbon with values of 1595cm^{-1} and 1359cm^{-1} respectively. The higher peak which shows the graphitic phase, shows that the material is highly graphitized. The morphology of the cassava peel derived activated carbon was determined by the scanning electron microscope (SEM) analysis and as displayed in Figure 4, it can be seen that the activated carbon consists of both micropores and mesopores. The inset of SEM micrograph is the elemental dispersive spectra (EDS) which display the elemental composition of the material as further seen in Table 2 with carbon content showing the highest peak, as the most present composite.

A Nitrogen adsorption/desorption analyser was used to get a better understanding of the pore structure of the carbon material. In Figure 3, the adsorption isotherms from the beginning corresponds to micropore adsorption(Xie et al., 2020). It can be noticed that a type IV hysteresis loop is formed later on showing the presence of mesopores. The sample shows adsorption-desorption sharp curve at a relatively low pressure while a hysteresis loop is observed at P/P_0>0.45, which indicates the coexistence of mesoporous and microporous structures (Amakoromo, Abumere, Amusan, Anye, & Bello, 2021). The KOH activated carbon showed a well-developed porous architecture having a BET surface area of 828 m^2g^{-1} (Table 3), the total mesopore and micropore volume otherwise known as the total pore volume was estimated to be 0.3981m^3g^{-1} and having a pore diameter of 2.66nm.

In Table 4, a comparison is made with other precusors and even cassava peels on the achieved surface area as reported by various researchers. The results achieved from this research clearly outperform the others in terms of specific surface area.

Table 4: Comparison of Specific surface area of different materials

Materials	**Activating agent**	**Specific surface Area (m^2g^{-1})**	**Ref**
Waste tires	H_3PO_4	563	(Zhi et al., 2014)
Petals	Physical (CO_2)	509	(Yu et al., 2017)
Rubber seed shell	KOH	620	(Pagketanang et al., 2015)
Cassava peel	$FeCl_3$	405.9	(Ndongo et al., 2020)
Cassava peel	KOH	828	This work

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CONCLUSION

The results achieved clearly show that cassava peel derived activated carbon can be used for multiple purposes. KOH activation also proves better than $FeCl_3$ as it delivers greater surface area and allows for better pore volume for application as a good adsorption material. Its porosity and high surface area can also be explored for energy storage applications.

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