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Several streams of agricultural residue is produced during agricultural activity. Activated carbon is a highly porous carbon material that is known to have good adsorption capacity. Rice husk, due to its high cellulose and lignin content, can be used to prepare activated carbon. The potential of rice husk was studied for the production of highly surface area activated carbon. The activated carbons were prepared via chemical activation with phosphoric acid and zinc chloride to identify the most suitable activating agent. The obtained activated carbons were characterized using scanning electron microscopy, BET surface area, and energy dispersive spectrometry (EDS). Phosphoric acid activation produced activated carbon with the highest porosity and surface area (427.154m^2/g) compared to that by zinc chloride activation. Carbon was the most dominant element observed in the prepared activated carbons, with the highest percentage of 85.67 % was shown by phosphoric acid activation.

Keywords: rice husk; activated carbon; chemical activation.

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ABSTRACT

Several streams of agricultural residue is produced during agricultural activity. Activated carbon is a highly porous carbon material that is known to have good adsorption capacity. Rice husk, due to its high cellulose and lignin content, can be used to prepare activated carbon. The potential of rice husk was studied for the production of highly surface area activated carbon. The activated carbons were prepared via chemical activation with phosphoric acid and zinc chloride to identify the most suitable activating agent. The obtained activated carbons were characterized using scanning electron microscopy, BET surface area, and energy dispersive spectrometry (EDS). Phosphoric acid activation produced activated carbon with the highest porosity and surface area (427.154m²/g) compared to that by zinc chloride activation. Carbon was the most dominant element observed in the prepared activated carbons, with the highest percentage of 85.67 % was shown by phosphoric acid activation.

Keywords: rice husk; activated carbon; chemical activation.

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I. INTRODUCTION

Agricultural activity is one of the occupational practices in most of sub - Saharan Africa. As a result, huge amount of waste products are generated yearly, which could be potent resources for sustainable energy sources (Yakovlev *et al.,* 2015). The high interest in a bio - based economy has stimulated a fast growing research and development on the biomass conversion techniques (Gabriele *et al.,* 2011). Activated carbon production from agricultural waste as possible economic and environmental effects. Activated carbons are used for the conversion of agricultural waste to adsorbents to remove organic chemicals, heavy metals from water which are of economic importance. Production of activated carbon from waste agricultural residue will minimize its importation (Ekpete *et al.*, 2016).

Latest developments in the preparation of carbon electrodes using several biomass residues for use in energy storage devices, such as batteries and supercapacitors are recently embraced by scientist as well researchers. The biomass residues usage as the primary precursor for the production of carbon electrodes has been increasing over the last years due to it being a renewable source with comparably low processing cost, providing prerequisites for a process that is economically and technically sustainable (Simões dos Reis *et al.*, 2020).

Liu *et al.* (2015) prepared highly surfaced-area activated carbon from coconut shells using KOH as activating agent. The activated carbon was used as a battery cathode. Similarly, Osman *et al.* (2016) synthesized activated carbon using different parts (shaggy and core) palm oil empty fruit bunch through the physical activation (at temperature of 600, 700, and 800°C). The result showed that the highest yield for activated carbon was achieved at 700°C. Dieu *et al.* (2020) prepared activated carbon for high - performance supercapacitors using oil palm empty fruit bunches (EFBs), through series of cleaning, carbonization and chemical activation processes. The activated carbon has a specific surface area of 2774 m²/g, which is very high among AC synthesized from the biomass materials. The specific capacities of AC and nitrogen-doped AC are found to be 182 and 217 F/g, respectively. This

was determined at the current density of 0.5 A/g using 6M KOH as aqueous electrolyte.

Optimization of Activated carbon preparation from Palm empty fruit bunch fiber process was studied by Arshad *et al.* (2019) using RSM-design under CO_2 atmosphere where the effect carbonization temperature and time were studied. The maximum I_2 adsorption of 373.41 mg/g with surface area (BET) of 1.09 m^2/g was observed at the end of the study. The results show that optimal carbonization condition for the process is achieved at 450°C for 90 min. Also maximum I_2 adsorption of 841.32 mg/g was achieved. The BET surface area of 900.02 m^2/g also was achieved. The activated carbon percentage yield of 49.33% was obtained at 500°C carbonization temperature by Hanum *et al.* (2017) from dried rice husk carbonized from 400 to 600°C different temperature under constant nitrogen atmosphere. After that, the carbon was treated with hydrochloric acid.

Activated carbon production was done mainly via two routes; first, through carbonization of the biomass under inert or oxidized condition. Second, through physical activation or chemical activation at lower temperature which gave a better quality of porous structure ACs and lower energy costs (Ioannidou and Zabaniotou, 2007). Prahas *et al.* (2008) reported that widely used chemical reagents for activation are $ZnCl_2$, NaOH, H_3PO_4, and metal chemicals (e.g., KOH and K_2CO_3). This Chemical activation is a commonly used method as reported in the literature (Lillo-Ródenas *et al.,* 2003; Kalderis *et al.*, 2008 and Adinata *et al.*, 2007). A high surface area up to 700~2000 m^2/g activated carbon is expected when the biomass waste is used as feedstock and chemical activation a production method. The properties of the obtained carbon materials strongly depend on the structure determined by the type of starting material, activating agent, and process conditions (Hesas *et al.,* 2013).

Presently, researchers are searching for an alternative and benign approach to producing the most low-cost activated carbon as an inexpensive adsorbent for the treatment of wastewater from industrial waste. This study intends to determine the potentials of native rice husk samples to prepare activated carbons using Phosphoric acid and Zinc chloride as activating agents.

II. METHODOLOGY

2.1 Sample Collection and Treatment

Sample was procured from the Attajiri rice mill at Kware local government area, Sokoto. The AOAC (1999) analyses methods (with amendment) were employed to obtain a required sample for these analyses. The collected sample was washed with distilled water to remove debris, dust, and other impurities and then dried in a hot air circulating oven at 105˚C overnight to remove moisture. The dried sample was ground into powdered and sieved to a size of 4mm. Standard procedures were employed for the preparation of solutions and other reagents using deionized water.

2.2 Preparation of Activated Carbon

The activated carbons were prepared following a method described by Anisuzzaman *et al.* (2015) with some amendments. The dried rice husk sample was impregnated directly with H_2PO_3 reagent and labeled RHP, while impregnation with $ZnCl_2$ was labeled RHZ. After the soaking, the samples were kept for 72hrs with constant shaking to ensure sufficient absorption of the reagents. Then it was filtered and dried in an oven at 110°C overnight. After that the dried sample was semi carbonized in a Muffle furnace for 15 minutes at 200°C and then heated to a temperature of 500°C for 45 minutes.

After the chemical activation, the activated carbon was refluxed with distilled water for 3h at a temperature of 80°C to remove excess reagent. Then, reflux was repeated several times until the constant pH value was achieved. The activated carbon prepared was refluxed with 0.1M HNO_3 for 1h to remove heavy metals and ash.

2.3 Characterization of Activated Carbon

The activated carbons prepared were characterized using surface Analyzer and Scanning Electron Microscope to determine micropore size, micropore volume, BET surface area, and morphological properties of the carbons.

2.4 Morphology and Elementals composition of the activated carbon

Scanning Electron Microscope (SEM) was used to observe the surface morphology and porosity of the activated carbons. The SEM images of the activated carbon prepared were taken using JEOL JSM-5000V Scanning Electron Microscope. The EDS analysis was carried out for elemental composition of the samples determine.

2.5 Surface Analyses of the Activated Carbon

The surface area and pore volume of the Activated Carbons (AC) were determined using Quantachrome Instrument (Version 11.03) by N_2 adsorption isotherms at 77.35K. The AC were degassed prior to the analysis for 3 hours at 250 ^{0}C in a vacuum. The surface area was determined using Multipoint Brunauer-Emmett-Teller (BET) method, while pore volume and pore size were estimated by application of the Barrett-Joyner-Halenda (BJH) method. The DR method was used to determine micropore (S_{mi}) surface area.

III. RESULTS AND DISCUSSION

3.1 Porous Properties of the Activated Carbon

Table 1: Activated Carbon Porous properties

Sample	S_{BET}(m^2/g)	S_{mi}(m^2/g)	S_{mi}/ S_{BET}	S_{mi}/ S_{BET} (%)
RHZ	362.315	390.237	1.077	107.7
RHP	427.154	428.802	1.003	100.3

The porous properties of the prepared AC from rice husk are presented in Table 1. The BET surface area for RHZ is 362.315m^2/g and 427.154m^2/g RHP. As evident from the table, phosphoric acid activation gives a high surface and pore volume (Figure 1). Phosphoric acid-treated activated carbon showed approximately 8% higher BET surface area than Zinc chloride treated activated carbon. The creation of pores in the carbon may be attributed to the release of volatiles from the biochar on carbonation. The H_3PO_4 reacting within the cellulose structure is assumed to have induced an enhancement of the pore volume (Benadjemia *et al.*, 2011).

The pore size distribution is an important clue revealing the adsorption mechanisms of porous substances. Figure 2 shows the pore size distribution of the rice activated carbon. The pores of adsorbing material were divided into three categories; micropore (diameter < 2 nm), mesopopore (2–50 nm), and macropore (>50 nm) (Ryu *et al.*, 1999). It could be seen from (Figure 2) that rice husk AC has narrow pore size distributions, including both micropores and mesopores. Most pores of activated carbons were composed of mesopores with sizes ranging from 2 – 2.8 nm.

ACs also contained micropores with narrow range from 1.7 to 1.9nm. The shared porosity in the activated carbon was approximately 63.6% and 36.4%, mesopore and micropore sizes, respectively.

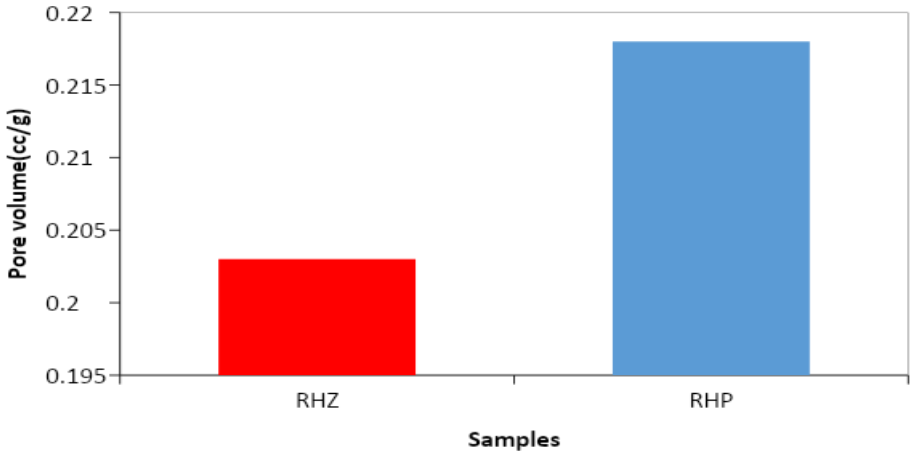

Figure 1: Pore size distribution of the activated carbons

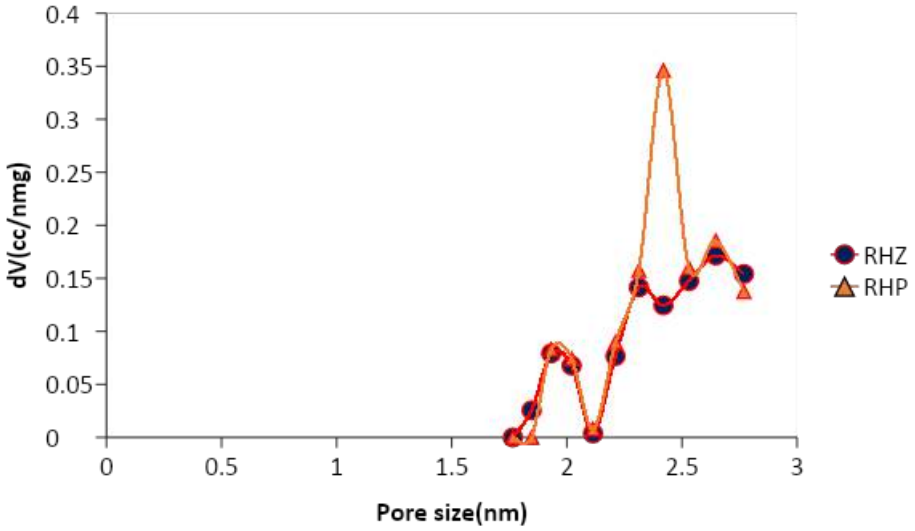

Figure 2: Pore size distribution of the activated carbons

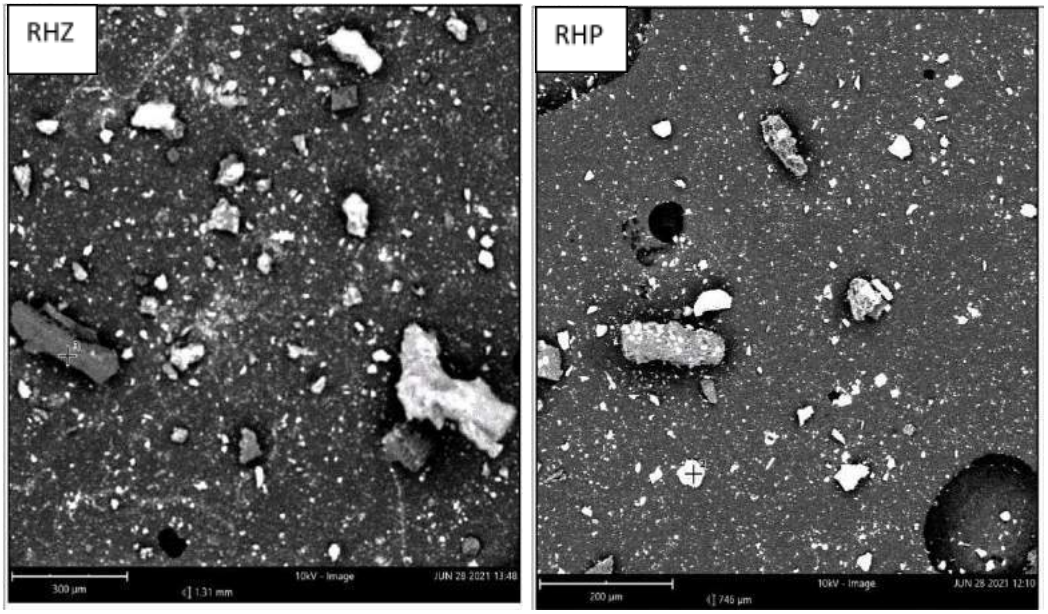

Figure 3: Scanning Electron Microscope (SEM) micrographs for ACs

Scanning electron microscopy (SEM) is an instrument for surface studies of adsorbents (Aljeboree *et al.*, 2017). It can also be used in the characterization of activated carbon. Evidence of porosity could be observed from the SEM images. The porosity was due to the decomposition of lignin, cellulose, and hemicellulose during carbonization, resulting in micropores and mesopores. The SEM structure in Figure 3 indicated that the porous structures were obtained on the activated carbons via chemical activation with $ZnCl_2$ and H_2PO_3. Also, it was observed that the surfaces of the activated carbon possess irregular circular pore structures and aggregation in different sizes and shapes. Phosphoric acid activation shows a better-cleared surface area compared to zinc chloride activation process. A well-clear surface of activated carbon may be attributed to the removal of impurities and volatile substances during activation. The presence of these porous structures implies the potential use of the prepared carbons as catalyst support because such pores provide sites on which active materials could be adsorbed to form an active catalyst

Table 2: Elemental Composition of the activated carbon

Sample	Carbon	Oxygen
RHP	85.67	14.33
RHZ	68.35	31.65

Elemental components of the prepared activated carbon presented in the Table 2 and EDS spectra (Fig. 4 & 5). Phosphoric acid-derived activated carbon (RHP) has a high carbon content of 85.67% and low oxygen content of 14.33 % compared to 68.35% and 31.65% for zinc chloride-derived activated carbon (RHZ). This indicates that phosphoric acid performs better as an activating agent than zinc chloride. This is also evident from the SEM image of RHP, which shows a better porous structure than RHZ.

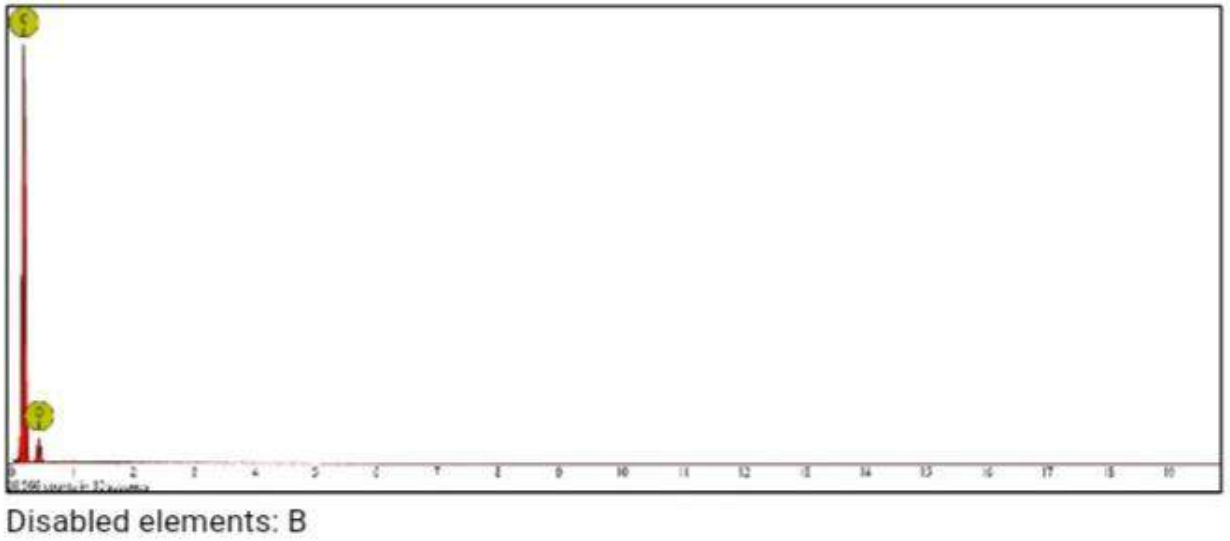

Figure 4: EDS spectra of RHP activated carbon

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IV. CONCLUSIONS

The present research studies compare phosphoric acid and zinc chloride as activation agents for the preparation of activated carbon from rice husk. The results show that AC prepared from Phos-

phoric acid activation has a higher surface area, percentage carbon, and better porosity compared to Zinc chloride activation. The results established that the development of porosity in the prepared carbons can be affected by two different agents. From this study it is evident that pore size distributions of the activated carbon could be achieved when different type of activation reagent are used.

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