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Preparation and Characterization of Activated Carbon derived from *Typha latifolia* (Cattail Grass) using H_3PO_4 and KOH as Impregnating Agents

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Abstract

In this study, activated carbons were successfully prepared from typha latifolia. The Activated Carbons were produced using potassium hydroxide (KOH) and phosphoric acid (H_3PO_4) as the impregnating agents and carbonized for 1h in a flow of N_2 gas at 700°C using tubular furnace. The Activated Carbons were analyzed by various characterization techniques such as Brunauer-Emmett-Teller (BET), Scanning Electron Microscope (SEM), Fourier Transform Infrared spectroscopy (FTIR), Thermo-gravimetric Analysis (TGA), and X-ray Diffraction (XRD) techniques . The BET results shows that the activated carbon impregnated with H_3PO_4 gives a surface area of 1083.733 m^2/g with a pore volume of 0.988 cc/g, followed by KOH activated carbon with surface area value of 501.999 m^2/g with a pore volume 0.253 cc/g. Lower surface area (352.490 m^2/g)with a pore volume (0.217 cc/g) were observed from the untreated activated carbon. The SEM micrographs of the activated carbons (Acs) revealed a heterogeneous surface structure with different pore sizes. The XRD analysis results depicted the amorphous carbon structure of the produced Acs with a broad peak at around 2θ = 25, 26 and 27 degrees. The TGA shows a major weight loss of about 73% for TLP-AC and 81% for TLK-AC. The functional groups present on the Acs were determined by FTIR analysis and the presence of carbonyl and hydroxyl groups confirms the presence of the anticipated functional groups in the as-produced materials.

Keywords: Activated Carbon, *Typha latifolia,* KOH, H_3PO_4, Carbonization

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INTRODUCTION

Activated carbon, also known as activated charcoal or activated coal, is a form of carbon materials that are distinguished by their extremely large specific surface areas, well-developed porosity, and tunable surface-containing functional groups (Baker *et al.,* 1992). Activated carbon is a versatile adsorbent widely employed in various industrial applications, such as water purification, air filtration, and gas separation (Ibsa *et al*., 2022). Activated carbon (AC) is a carbonaceous substance made by pyrolyzing a variety of low-cost materials with a high carbon content and low inorganic content (Boadu *et al*., 2020). Activated carbon is generated on a commercial scale by pyrolysis and activation of high-cost starting materials such as wood, petroleum, and coal, rendering it costly for pollution control (Alothman *et al*.,2011). The use of sustainable and less costly precursors for the preparation of activated carbon is attracting the attention of researchers all over the world as a way to minimize production costs. Activated carbon can be produced from variety of industrial and agricultural by-products, as well as forest wastes including coconut shell, sugar beet bagasse, rice straw, bamboo, rattan sawdust (Reyam *et al*.,2018; Asha *et al*., 2021), molasses (Hameed *et al.,* 2007), rubber wood sawdust (Prakash *et al*,2006), oil palm fiber (Tan *et al*., 2007), waste apricot, coconut husk and other biomass (Radhika *et al*., 2006; Rhaman *et al*., 2015). *Typha latifolia is* commonly known as cattail and Kachala by the Hausa people who live around the Hadejia-Nguru wetlands regions of Northwestern-Northeastern Nigeria. The presence of large quantities of Typha latifolia grass has posed a lot of challenges to the people living around the wetland areas. Its detrimental effect to the environment encourages researchers to produce AC from the grass. It is believed that *Typha* grass entered Nigeria's inland marshes from East Africa. *Typha latifolia* is the most widely distributed species, occurring in most of North America, Europe, Asia, and Africa (Zungum *et al.,* 2019). *Typha Latifolia* is a prolific wetland plant known for its wide distribution and rapid growth (Teshome *et al*., 2020; Lawan *et al*., 2023, Mohammed and Rabiu, 2022). The plant possesses unique characteristics, including a fibrous structure and high carbon content, making it a potentially valuable raw material for activated carbon production (Sanchez-Orozco *et al*.,2018; Elkhatib *et al*., 2019). Moreover, the use of *Typha latifolia* aligns with the principles of green chemistry and sustainability, as it is abundant in many regions and can be harvested without causing ecological harm. As the demand for sustainable and cost-effective adsorbents rises, investigating the production and characterization of activated carbon derived from *Typha latifolia* becomes imperative. This study contributes to the field by exploring the feasibility of utilizing a readily available, less expensive and environmentally friendly biomass for the production of activated carbon, addressing both the need for alternative feedstocks and the sustainability challenges associated with convectional sources and comparing the two AC.

The utilization of *Typha Latifolia* as a raw material for activated carbon production holds promises due to its abundance in wetland ecosystems and its potential to provide an environmentally friendly alternative to conventional sources. The efficient conversion of *Typha latifolia* into activated carbon involve physical and chemical activation processes (Babatunde *et al*., 2021). The physical activation process typically involves the controlled pyrolysis of the biomass under inert conditions, resulting in the development of a porous structure with enhanced surface area (Musa Sania and Abdullahi Muhammad Ayubaa, 2022).On the other hand, chemical activation employs activating agents, such as KOH or H_3PO_4, to create pores and increase the carbon's reactivity(Asmamaw *et al*., 2023).The selection of activation method plays a critical role in determining the textural and chemical properties of the derived activated carbon (Ibsa *et al*., 2022; Shahcharagh *et al*., 2023). Characterizing the properties of *Typha latifolia*-derived activated carbon involves a comprehensive analysis to assess its suitability for specific applications includes; Brunauer-Emmett-Teller (BET) analysis, Scanning electron microscopy (SEM), Fourier transform infrared (FTIR) spectroscopy and X-ray diffraction (XRD) (Liu *et al*., 2023). The

primary objective of this work was to prepare activated carbon derived from *Typha latifolia* using KOH and H_3PO_4 as impregnating agents and its characterization for surface area, pore volume and pore size distribution of the as-prepared activated carbon and to advance the understanding of the influence of different impregnating agents on the properties of activated carbon. By comparing the effects of KOH and H_3PO_4 and to identify the most effective agent for optimizing the performance of activated carbon derived from *Typha latifolia*, thereby contributing to the development of sustainable and efficient adsorbent for various environmental and industrial applications.

MATERIALS AND METHODS

Sample collection and preparation

The *Typha Latifolia* used for this study was obtained from a wetland in the Hadejia Local Government region of Jigawa State using a straight forward random sampling procedure. Following the removal of the dirt from the grass sample, it was rinsed with tap water, followed by distilled water, dried in an oven at 60°C, then grinded into a powder using a mortar and pestle.

Preparation of Activated Carbon

Impregnation was done according to method described by Nasir, *et al*., (2018). About 10 g of grinded grass sample was impregnated with 100 mL of 5 M H_3PO_4 for 24 hours. The same amount and concentration of KOH was also used to impregnate 10 g of grinded grass for 24 hours. After impregnation, the wet samples were dried at 110°C for 12 hours in an oven.

The samples were carbonized using tube furnace at 450°C under a nitrogen atmosphere at a constant heating rate of 10°C/min for 1 hour and then cooled at room temperature in a desiccator. The resulting activated carbons were washed with deionized water till filtrate becomes neutral (pH 7). These neutral activated carbons were then dried in an oven at 110°C until all of the moisture from the infusion is gone.

The neutral dried carbonized samples were further activated in a furnace set at 700°C under a nitrogen atmosphere with a steady heating rate of 10°C/min for 1 hour and then cooled at room temperature in a desiccator. The activated carbons produced were stored in plastic sample container before further characterization.

Characterization of Activated carbon

Fourier Transform-Infrared Spectroscopy (FT-IR, Buck M530, USA) was used to examine the surface functional groups of the prepared activated carbons and were scanned in the range of 650 – 4000 cm^{-1}. Scanning Electron Microscopy (SEM Phenom ProX, by Phenom World Einhoven quorum technologies model Q150R) was used to determine the surface morphology of the activated carbons. X-ray Diffraction (XRD) (Rigaku Miniflex Goniometer) was used to identify the nature of the crystalline and the amorphous phases in the activated carbons. Thermogravimetric analysis (Thermogravimetric Analyzer, PerkinElmer MSE-TGA 4000, Netherland) was used to evaluate the decomposition temperature of the carbonized activated carbons and BET (Quantachrome NOVA 2200E, USA) was used to investigate the surface area and pore size distributions of the activated carbons respectively.

RESULTS AND DISCUSSION

BET Surface Area

The results of BET surface area (m^2/g), pore volume and pore size distribution are presented in Table 1. The *Typha latifolia* phosphoric acid-based activated carbon (TLP-AC) sample has surface area of 1083.733 m^2/g, a pore volume of 0.988 cc/g and pore size distribution of 2.9 nm while *Typha latifolia* potassium hydroxide –based activated carbon (TLK-AC) has surface

area of 501.999 m^2/g, pore volume of 0.253 cc/g and pore size distribution of 2.1 nm, whereas the untreated (TLo-AC) shows lowest surface area, pore volume and pore size distribution of 352.490 m^2/g, 0.217 cc/g and 2.1 nm respectively. This result is in agreement with the report of Evwierhoma, *et al.*, (2018) who found that the activated carbon prepared using H_3PO_4 has highest surface area. The result also shows that the surface area of the activated carbon samples treated with impregnating agent increases as the concentration of the impregnating agents' increases, as seen from the BET study which is consistent with work of Ephraim *et al.,* (2017).

The prepared ACs were found to be mesoporous and microporous as shown in Fig.1, this is as a result of the pore size distribution of 2.9, 2.6 and 2.1 nm respectively. And the highest pore size distribution of 2.9 nm accounted for TLP-AC where as TLo- AC has the lowest pore size distribution of 2.1 nm. Activated carbons are known to have a heterogeneous pore structure, which according to IUPAC is classified as micropores (<2 nm), mesopores (2-50 nm) and macropores (> 50 nm) (Jegi and Mkayula, 2002). Studies by Nasir et al., (2018) on nitrogen gas adsorption BET analysis found out that the most widely used industrial activated carbon has been shown to have a specific surface area ranging from 800 to around 1500 m^2/g. The findings of this study demonstrated that *typha latifolia* is suitable raw material for the preparation of activated carbon owing to its high surface area.

Table 1. The effect of different activation method on BET surface area of the as-synthesized activated carbons

Samples	Surface Area (m^2/g)	Pore Vol. (cc/g)	Pore Size (nm)
TLo- AC	352.490	0.217	2.132
TLP-AC	1083.733	0.988	2.920
TLK-AC	501.999	0.253	2.600

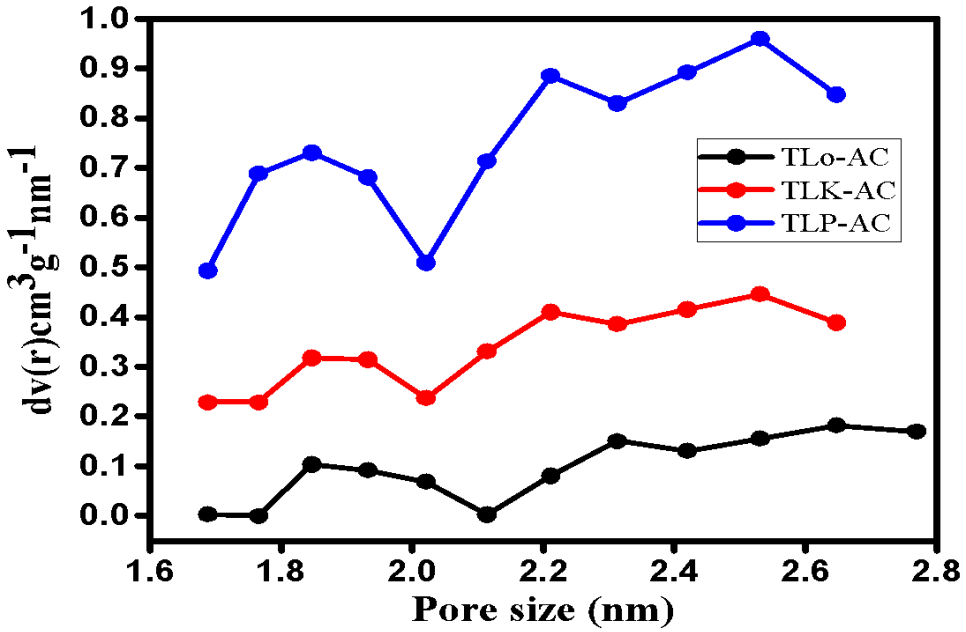

Figure 1: Pore size distribution of activated carbon

Scanning Electron Microscope (SEM)

The prepared activated carbons were examined using Scanning Electron Microscope (SEM). The SEM micrographs shown are spongy in their structure. The SEM micrographs of the chemically activated Carbon by H_3PO_4 and KOH were presented in Figure 2.

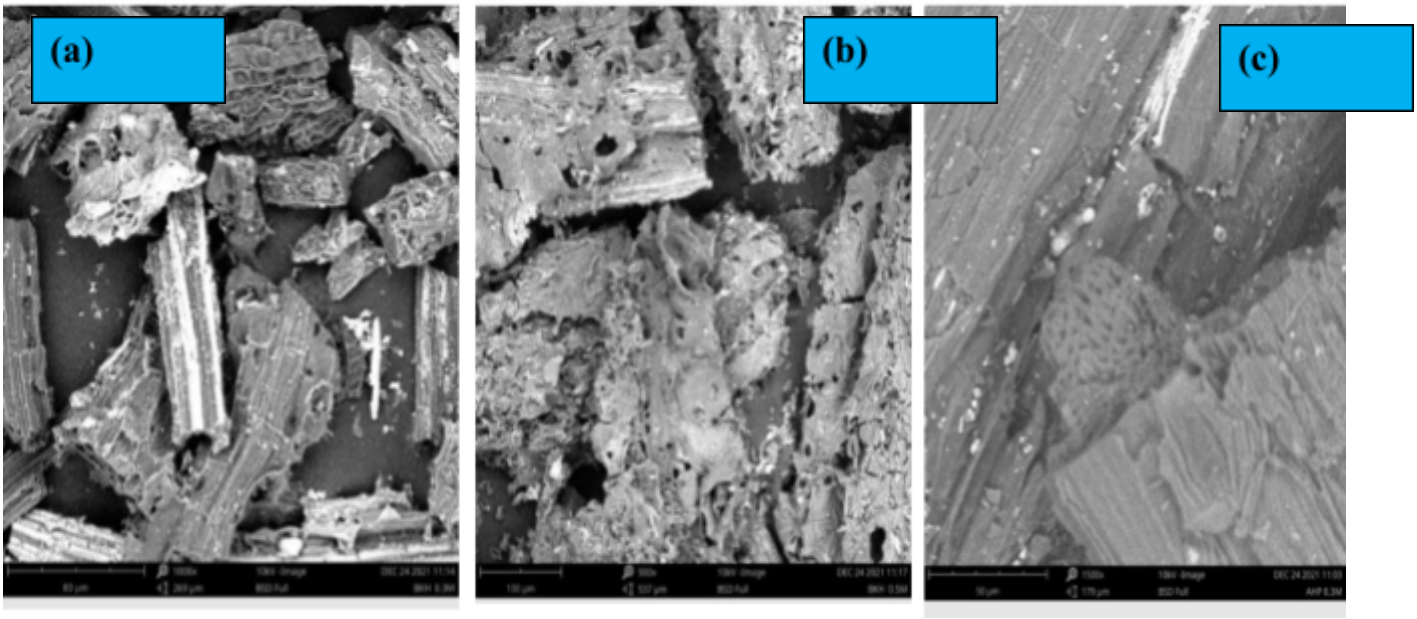

Figure 2: SEM Micrograph of activated carbon samples (a) TLK-AC (KOH-Based AC), (b) TLP-AC (H_3PO_4), and TLo –AC (Un-impregnated AC).

The ACs shows heterogeneous surface morphology with a well-developed porosity structure and pores with various sizes and shapes (Fig. 2). The activated carbons outer surface was incredibly uneven and filled with cavities as shown (Fig.2). These cavities were caused by the chemical reagent (H_3PO_4 and KOH) evaporation during carbonization (El-Hendawy *et al.,* 2008). The surface structures of the TLo - AC contain hazy pores, but in the TLK and TLP- ACs surface structures were clear, burnt-out pores. The shape of the pore increases as the impregnation agent concentration increases as shown by the micrograph. This report agrees with the observation made by Anisuzzamam *et.al.,* (2015) that high concentration of the impregnation agent has a positive effect on the average number of pores.

X-Ray Diffraction Analysis

The activated carbons prepared were subjected to X- ray diffraction analysis as shown in Fig.3. The X-ray diffraction patterns for the TLo -AC, TLK-AC and TLP-AC demonstrated the non-appearance of strong and sharp peaks, which denotes a high degree of disorder which is one of the characteristics of carbonaceous materials as reported by Ephraim *et. al.*, (2017). This indicates the predominance of amorphous carbon structure in the sample which is accompanied by a broad, low-intensity diffraction peak between 2θ = 20 and 30 degrees. The sharp peaks observed at 29.5 degree of TLP-AC may be due to the presence of phosphorus-containing groups interacting with other functional group (Ramakrishna, 2012).

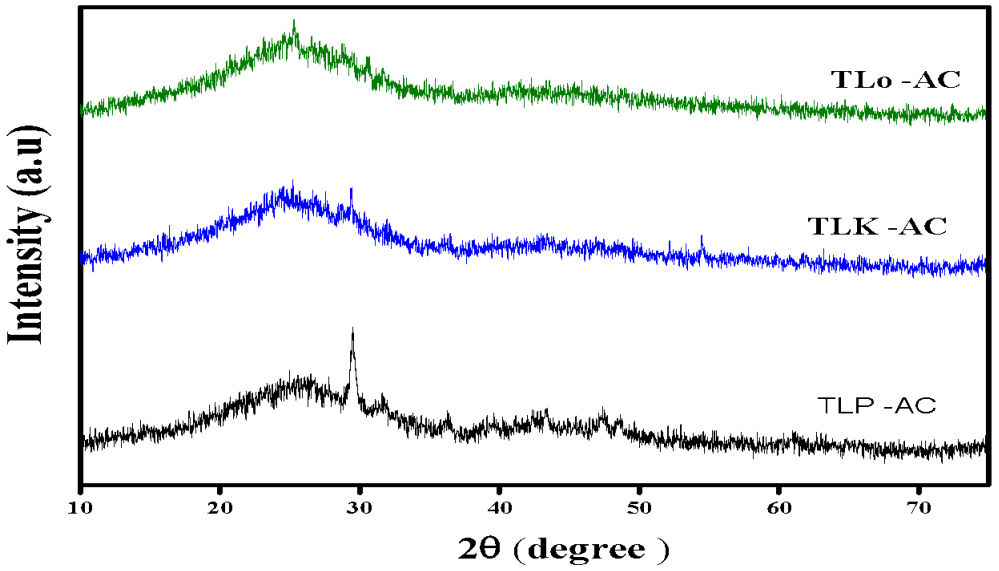

Fig. 3. X-ray diffraction pattern for the different ACs

Fourier Transform Infrared Spectroscopy (FT-IR)

The functional groups O-H, C-H, C=O, C≡C and C-O bands were observed at 3903, 2836 1686, 1169 and 1075 cm^{-1} respectively. A broad band from 3903 to 3265 cm^{-1} corresponds to the stretching vibration of hydroxyl group. The peak at 2836 to 2571 cm^{-1} indicate the presence of C-H stretching group. The peaks at 1686 cm^{-1}, 1169 cm^{-1}, and 1075 cm^{-1} corresponds to the C=O stretching, bridge C≡C stretching, and C-O stretching respectively. The peak around 854–690 cm^{-1} could be caused by the stretching of the P=O bond in phosphate ester, O–C bond in P–O–C linkage, or P=OOH bond. According to Anisuzzaman *et. al.*, 2015, the presence of this band indicates the presence of phosphorus-containing groups in the prepared AC. Thus, the appearance of distinct hydroxyl and carbonyl group peaks in the activated *Typha latifolia* indicates that the desired functional groups are present. The findings of the FT-IR study show that only very slight variations between the samples could be found. However, the TLP-AC and TLK-AC samples' shifting of bands and variations in wavelength numbers and absorbance suggest that chemical treatment likely resulted in chemical transformation.

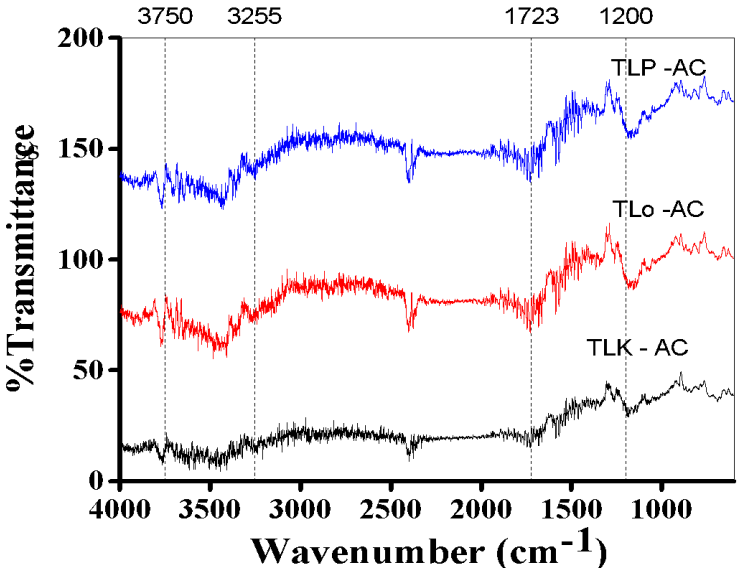

Fig.4. Fourier transform infrared (FTIR) spectra of various activated carbon prepared.

Table 2. FT-IR Spectra band assignment for sample ACs

Band Position (cm^{-1})	Suggested Assignment
3903 – 3265	C-H; O-H
2836 – 2571	O-H; C-H; S-H
2392	C≡N; C≡C
1686 – 1341	C=C; C=O; C=N
1169 – 1075	C-O
854 – 690	P=O

Thermogravimetric Analysis (TGA)

This analysis serves as the basis or initial guideline for determining the exact temperature range in which the activation is to be carried out. Fig 5 shows the TGA micrograph of TLK-AC and TLP-AC.

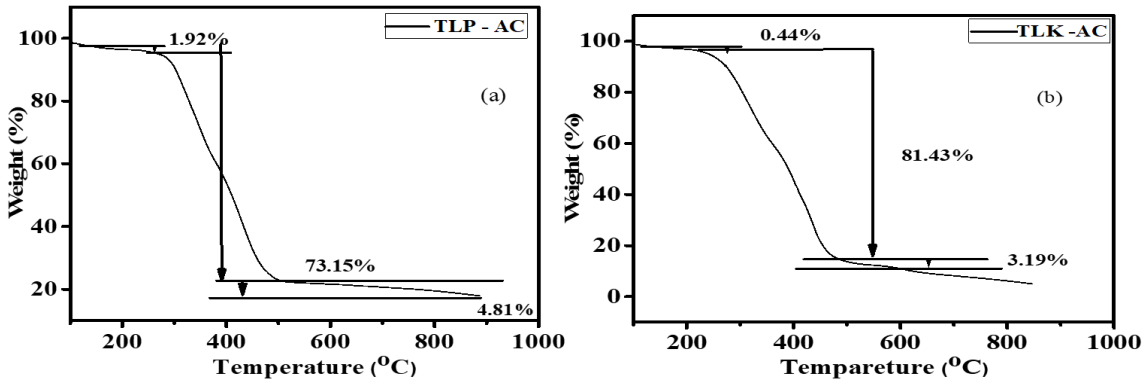

Fig. 5 TGA Curve for (a) TLP-AC and (b) TLK –AC

The results showed that a broad peak is observed between 10 and 100°C, which indicate the initial weight loss that occurs due to evaporation and the release of bond water molecules. This observation agrees with the report of Soni *et al.,* (2015). The TGA, micrograph for samples TLK-AC and TLP-AC showed weight losses of 81% and 73%, respectively. According to the TGA micrographs shown in Fig.5, the descending TGA thermal curve indicate that a weight loss occurred. Impurities, volatile matter, and moisture are all evaporated from samples of TLK-AC and TLP-AC between 90 and 150°C. Below this temperature, weight loss is only about 10%, and no significant de-volatilization occurs until temperatures between 200 and 450°C. From 350°C, the weight loss was gradual and continued up to 900°C, and a char is formed along with the partial formation of tar and the evolution of gases like CO, CO_2, or H_2. At higher temperatures, conversion of carbon to carbon based oxide occurs, resulting in the formation of condensable hydrocarbons. In the temperature range of 30-950°C, weight losses of 81% for sample TLK-AC and 73% for sample TLP-AC were noted. The removal of water molecules and other volatile materials from the carbon in this study is most likely what caused the weight losses that were observed. This study provided important information that is essential for the production, handling, description, and use of activated carbon.

CONCLUSION

Activated carbons were prepared from the pyrolysis of *Typha Latifolia* at 700°C by chemical activation using H_3PO_4 and KOH as the impregnating agents. The surface area of the activated carbons prepared by chemical activation were found to be higher than untreated carbon. In the study, the preparation of AC was successfully accomplished by converting *Typha Latifolia* into valuable product. The activated carbon impregnated with H_3PO_4 have the highest surface area compared to that of KOH-based activated carbon. The study provided insight related to the utilization of *Typha latifolia* as a precursor in the preparation of

activated carbon, this will go a long way in providing a solution to mitigate the negative effects of *Typha latifolia* in wetlands and habitat restoration.

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