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Synthesis of Porous Carbons from the Valorization of Polyethylene Terephthalate Waste

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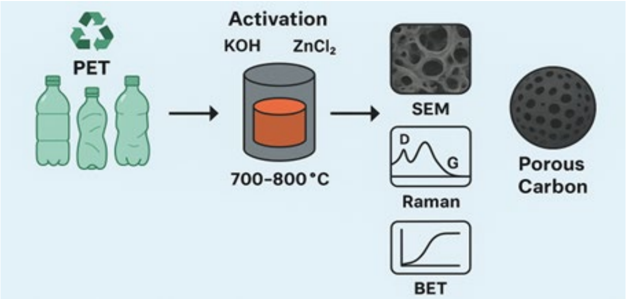
Abstract**: Upcycling plastic waste for water remediation is a promising paradigm for enabling a circular economy. This study aimed at investigating the synthesis of porous carbons from waste polyethylene terephthalate (PET) bottles using potassium hydroxide and zinc chloride as activating agents at temperatures ranging from 700 to 800°C. The influence of different activating agents and activation temperatures on the characteristics of porous carbons was investigated. The morphological and structural characteristics of the resultant porous carbons were analyzed using scanning electron microscopy (SEM), Raman spectroscopy, and nitrogen physisorption surface area analysis. The SEM data revealed interconnected porous morphologies for all the porous carbons obtained. Raman spectra showed two distinct D and G bands at 1336-1347 cm^{-1} and 1587-1599 cm^{-1}, respectively, indicating carbon graphitic structures. The I_D/I_G ratios revealed varying degrees of graphitization depending on the activation agent and temperature. The highest surface area obtained was 289 m^2g^{-1} at 800°C with porous carbons synthesized using KOH as the activating agent. This study demonstrates that waste PET is a viable and sustainable precursor for fabricating porous carbons.**

Keywords**: Porous carbons, Chemical activation, Waste polyethylene terephthalate.**



Graphical Abstract

1. Introduction

Porous carbons have versatile applications due to their characteristics, which include tunable porosity and surface area, presence of surface functional groups, tunable electrical and thermal conductivity, chemical stability, low density, and high mechanical strength [1]. Owing to their outstanding properties, porous carbons have been applied in gas storage, water purification, energy storage and catalysis, among other applications [2]. The synthesis of porous carbons involves the use of carbon-rich precursors from both natural and synthetic sources, including biomass, polymeric molecules, and carbonaceous waste materials. Carbonaceous waste materials are more desirable feedstocks owing to their affordability, and high carbon content. In the realm of carbonaceous waste materials, plastic waste, namely from thermoplastics, are a promising carbon feedstock in the synthesis of porous carbons due their ability to be pyrolyzed at higher temperatures (≥ 400 °C) depending on the polymer decomposition temperature[3]. In addition, they are readily available because of the massive utilization of plastic in various aspects of modern society. The surge in plastic use has been linked to extensive production of plastics for use especially in packaging, building and construction, automotive, electronics, agriculture, and medical applications [4]. According to the Organization for Economic Co-operation and Development (OECD), the annual production globally soared from 234 million tons in 2000 to 460 million in 2019. This resulted in approximately 353 million tons of plastic waste in 2019 which is double the amount produced in 2000 (156 million tons). The same report claimed that only 9% of this waste was recycled, while 19% was incinerated and approximately 50% ended up in sanitary landfills. The remaining 22% was dispersed into uncontrolled dumpsites, burned openly or leaked into the environment. In 2019 alone, 22 million tons of plastic leaked into the environment [5].

Fig. 1. Used PET bottles

Polyethylene terephthalate (PET) is a prevalent source of

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*Corresponding author: bridget@uonbi.ac.ke plastic waste, given its utilization in the fabrication of disposable water bottles, textiles, and packaging materials [6]. The disposable PET bottles that are released into the environment degrade over time and release microplastics which contribute not only to environmental pollution but also to the deterioration of human health through changes impacted on eutrophication, ozone layer depletion and water scarcity as well as particulate matter formation [7]-[9]. Thus, a plausible approach for their conversion into useful porous carbons is needed to address this issue. PET stands out as an ideal precursor for porous carbon synthesis due to its approximately 11% fixed carbon content, and low inorganic composition [10]. The process of synthesizing porous carbons from plastic waste typically involves a carbonation process where carbonaceous waste is converted into char by pyrolysis. Char is a highly carbonaceous material that can be further upgraded to functional materials through a pre- and or post-treatment process, otherwise known as the activation process [11]. The pyrolysis of PET yields products such as char, liquid oil and gases [12]-[14]. The activation process can be done by two means, physical and chemical activation [15]. In physical activation, the carbonized material is activated by heating at temperatures between 700-900 °C in the presence of steam or CO_2. Chemical activation on the other hand involves impregnating the carbonaceous material with a chemical, typically a concentrated acid or base, and then subjecting it to temperatures of between 400-700 °C in an inert environment [16]. The activation temperature can be from 400-1000 °C depending on the activating agents [17]. Chemical activation has an advantage over physical activation in that it produces higher yields and higher surface areas at lower activation temperatures and times [17], [18]. The properties of porous carbons are influenced by factors such as the precursors used, pretreatment of the precursor before carbonization, the chosen carbonization process as well as the activation process that is done before or concurrently with carbonization [19]. The activation process introduces a high surface area and a well pore organized network with various surface functionalities into the carbon skeleton [20]. As such, the porosity and surface area of porous carbon materials are highly dependent on the choice of a precursor and the parameters of the activation process. Although the upcycling of plastic waste to porous carbons by physical and chemical activation has been reported by other researchers [21], the effects of using the potassium hydroxide and zinc chloride as activating agents on the properties of the porous carbons have not been fully explored. Herein, we report the upcycling of waste polyethylene terephthalate (PET) into porous carbons by comparing the effects of using different activating agents (KOH and $ZnCl_2$) and varying the activating temperatures (700–800 °C).

2. Materials and Methods

A. Materials

Nitrogen gas, N_2 (99.99%) was purchased from Jcall, Kenya, Potassium hydroxide, KOH (85%), zinc chloride $ZnCl_2$ (97%) and hydrochloric acid HCl (37%) were all purchased from Loba Chemie and used without further purification.

B. Preparation of Porous Carbons

The PET waste used in this study was collected from household waste consisting of empty PET water and cold drink bottles. The PET waste bottles were cut into small pieces of about 8-10 mm, carefully washed with distilled water, and dried in a preheated oven at 60°C for 12 h. Then the PET waste was precarbonized at 600°C in a quartz tubular furnace under N_2 environment, with a heating rate of 10°C min^{-1} for 1 hour and labelled as precarbonized char. The precarbonized material was then mixed with KOH at a mass ratio of 1:3 (precarbonized char: KOH). Then the resulting mixtures were dried in an oven for 12 h at 105°C. The mixture was then transferred into a tubular furnace and heated up to 700°C at a heating rate of 10 °C min^{-1} under nitrogen gas. When the desired temperature was achieved, the system was kept isothermally for 1h and thereafter, the furnace was cooled to room temperature. Following this, the washing of samples was carried out with 1M hydrochloric acid and the pH of the solution neutralized by diluting with distilled water. Thus, excess KOH and chlorides in the samples were drained out. Lastly, the activated samples were dried in an oven at a temperature of 110°C for 12 h to obtain the porous carbons. This process was repeated for 800°C. The porous carbons were labelled as PCK700, PCK800 for the different activation temperatures of 700°C and 800°C, respectively. For comparison purposes, similar procedures were utilized on porous carbons activated using zinc chloride and the samples labelled as PCZ700 and PCZ800, for the different activation temperatures of 700°C and 800°C, respectively.

C. Materials Characterization

Scanning electron microscope (SEM) micrographs were carried out using a TESCAN VEGA LMS SEM. The SEM measurements were conducted at 1.0 kV accelerating voltage. Raman spectroscopy measurements were carried out to determine the degree of graphitization of the porous carbons using a Laser STR Raman spectrometer. Raman scattering was recorded using a laser of wavelength 532 nm with a maximum laser power of 2.25 W and an exposure time of 5 s. The nitrogen physisorption surface area analysis was performed by Brunauer-Emmett-Teller (BET) method using a Quantachrome Instruments Autosorb automated gas sorption analyser iQ. The N_2 adsorption isotherm was recorded at relative pressures reaching 1 atm, using ultrahigh purity N_2 gas (>99.999%). The measurements were carried out at a constant temperature of 77 K.

3. Results and Discussion

A. Morphological Features

Figure 2 depicts the microstructural morphology of synthesized porous carbons as shown by scanning electron microscopy (SEM). Figures 2(a) and 2(b) display porous carbons obtained after activation with potassium hydroxide (KOH), revealing a surface topography with notable fractures and fissures. This suggests successful activation of the carbon surface with KOH through the formation of an interconnected porous morphology. PET is composed of ethylene glycol monomers and terephthalic acid; during pyrolysis, the carbon chains are broken, and char is produced [13]. At 400 ˚C, KOH undergoes a reaction with carbon, yielding potassium carbonate. When temperatures exceed 700 ˚C, the potassium carbonate decomposes, releasing CO_2. This release of CO_2 promotes further carbon gasification, leading to increased porosity development [22]. Additionally, the breakdown of potassium carbonate generates metallic potassium, which intercalates into the carbon framework. During the washing process, metallic potassium is extracted from the carbon, widening the gaps between carbon atomic layers and augmenting pore volume [23].

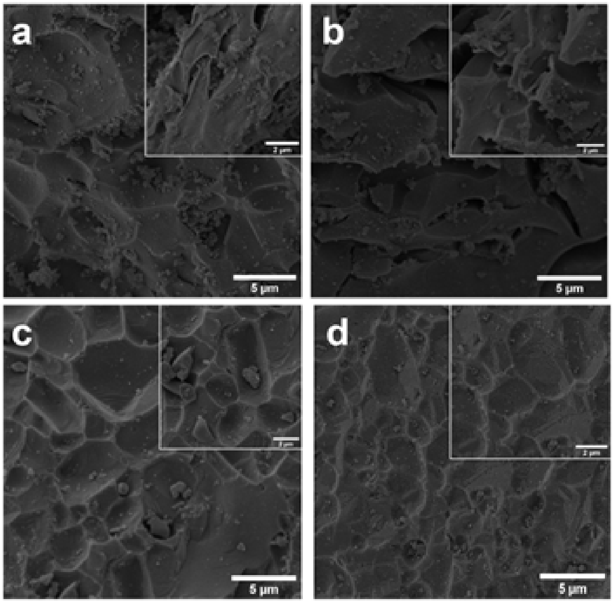

Fig. 2. Scanning electron microscopy (SEM) images (a) PCK700, (b) PCK800, (c) PCZ700, (d) PCZ800, respectively

Figures 2(c) and 2(d) show carbons synthesized using zinc chloride ($ZnCl_2$), which have a distinct morphology. Despite having an interconnected porous morphology, their surfaces appear to be more compact than their KOH-activated counterparts. Zinc chloride acts as a dehydrating agent when exposed to high temperatures, thus causing pores to form within the carbon structure [24]. During activation, the zinc chloride is intercalated into the carbon matrix at temperatures exceeding zinc chloride's melting point (732 °C), creating an expanded cross-linked structure with the carbon char [24]. The carbon atoms interact with the dehydrating agent and upon removal of the unreacted salt, a porous carbon is formed. The atomic size for potassium (1.38 Å) is higher than that of zinc (0.74 Å) and this could aid in explaining the slight difference in the morphology observed. A comparison of porous carbons formed at 800 °C to those obtained at 700 °C reveals a more porous morphology in the former due to the temperature being greater than the zinc chloride melting point and the breakdown of potassium carbonate. The chemical activation process aided in the formation of an interconnected porous morphology, agreeing with similar works in literature [25], [26].

B. Structural and Textural properties

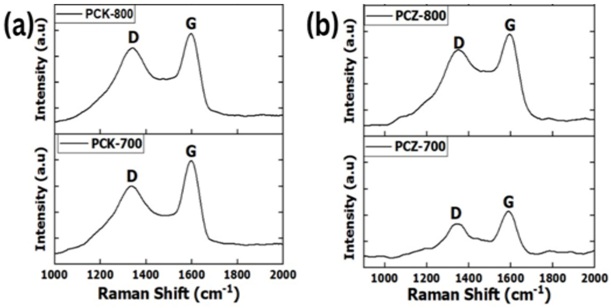

Fig. 3. Raman spectra for the porous carbons obtained from; (a) KOH-activating agent and (b) ZnCl2 activating agent, respectively

Figure 3 (a) depicts carbons activated through the utilization of potassium hydroxide (KOH), whereas Figure 3 (b) illustrates carbons activated using zinc chloride ($ZnCl_2$). The spectra show two distinct bands, the lower shift (1336-1347cm^{-1}) represents the D band, and the higher shift (1587-1599cm^{-1}) represents the G band. The presence of D peaks signifies the A_{1g} breathing mode of sp^2 carbons, while the G band indicates the presence of in-plane E_{2g} vibration mode of sp^2 carbons in graphite [27]. Similarly, previous studies have observed distinct D and G peaks in synthesized porous carbons [28], [29].

Table 1
Comparative D and G band positions and I_D and I_G ratios

Material	D band (cm^{-1})	G band (cm^{-1})	I_D/I_G ratio
PCK-700	1336	1599	0.76
PCK-800	1344	1595	0.86
PCZ-700	1340	1587	0.77
PCZ-800	1347	1595	0.85

Table 1 presents the I_D/I_G ratios which serve as indicators for the degree of graphitization of the carbon structure [30]. The I_D/I_G values are relatively higher in porous carbons synthesized at 800 °C, indicating the creation of structural defects and a lower degree of graphitization as compared to those obtained at 700 °C. The increase in the I_D/I_G ratio with rising activation temperature indicates that higher temperatures promote structural disorder and defect concentration, thereby lowering the degree of graphitization [27], [30]. In addition, the I_D/I_G ratios for the porous carbons synthesized using potassium hydroxide (KOH) were slightly higher in comparison to those prepared using zinc chloride. This suggests that KOH creates a more disordered, defect-rich structure, whereas $ZnCl_2$ facilitates a slightly more ordered and graphitized carbon matrix.

Figure 4 shows the nitrogen physisorption isotherms of the synthesized PET-derived porous carbons. According to the IUPAC classification of gas adsorption isotherms, the BET isotherms for PCK700 and PCK800 fit the type IV BET isotherm well. They also exhibit a pronounced type H4 hysteresis loop which indicates the presence of mesopores (pore diameter between 2-50nm) [31]. The PCZ700 and PCZ800 fit the type III isotherm [32], [33] this type of isotherm is characterized by a concave shape where there is a slow increase in the amount of nitrogen adsorbed at low relative pressures followed by a rapid increase at higher pressures without reaching a plateau. A type III isotherm indicates the presence of a broad distribution of pore sizes, with wider micropores and narrower mesopores. PCZ800 does not show hysteresis, a small hysteresis is however observed with PCZ700, which indicates the presence of mesopores.

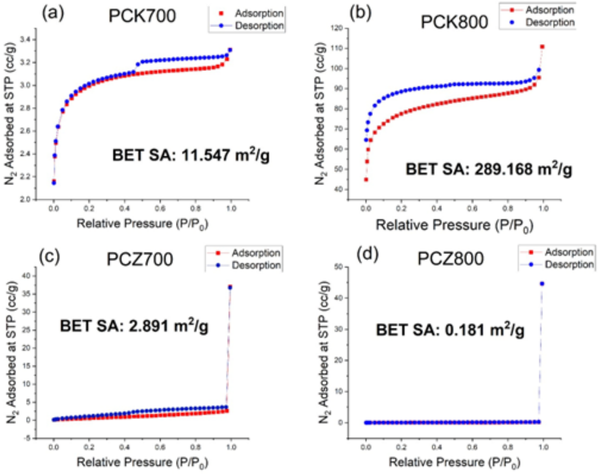

Fig. 4. N2 physisorption Isotherms (a) PCK700, (b) PCK800, (c) PCZ700 and (d) PCZ800, respectively

Porous carbons activated with KOH showed a higher surface area compared to those synthesized u sing zinc chloride as the activating agent. PCK800 had the highest surface area (289.168 m^2/g) while the lowest surface area was observed with PCZ800 (0.181 m^2/g). Activating the carbons at 700 ºC resulted in a higher surface area for porous carbons synthesized using zinc chloride. This corresponds to results obtained by [34] where there was a decrease in surface area for porous carbons activated using zinc chloride as activation temperature increased from 700 ºC to 800 ºC. This can be attributed to pore narrowing and collapse that occurs at higher temperatures due to sintering effect of the volatile compounds formed and the shrinkage of the carbon structure. A similar decrease in surface area was observed when temperature was increased from 500-900 ºC by [35], the highest micropore surface area was obtained at 700 ºC, and after that, a declining trend was observed. This was due to the excessive burn-off of carbon constituents at high temperatures. For porous carbons synthesized using potassium hydroxide, the surface area increased with increase in activating temperature. A similar observation was noted by [36] during chemical activation of biochar with KOH. An increase in the activation temperature from 600-900 ºC improved the surface area and porosity of porous carbons. During activation with KOH, K_2CO_3 is formed, and it further decomposes to form K_2O and CO_2. Increase in temperature facilitated the formation of K_2O, which disrupted the existing pores and led to their expansion, thus enhancing the pore structure of the carbons [36], [37]. The higher surface area values for the porous carbons obtained using KOH activating agents compared to those obtained from the porous carbons obtained from $ZnCl_2$ corroborates the Raman data and the SEM images.

4. Conclusion

Porous carbons were produced from PET waste via chemical activation employing zinc chloride and potassium hydroxide as activating agents. SEM images confirmed the successful synthesis of porous carbon with a porous interconnected morphology. The porous carbons showed degrees of graphitization that decreased with an increase in activation temperature for both $ZnCl_2$ and KOH activated carbons. The surface area of the porous carbons increased with increasing temperature for all the synthesized porous carbons. Raman spectroscopy demonstrated the effect of increasing the activation temperature on the structural properties of the porous carbons. The porous carbons obtained at 800 ºC exhibited higher I_D/I_G ratios for both $ZnCl_2$ and KOH-derived carbons. Moreover, the KOH-derived carbons consistently demonstrated slightly lower degrees of graphitization compared to their $ZnCl_2$ counterparts at similar temperatures, highlighting KOH's promotion of a more defective carbon structure. The most significant difference was noted in the textural properties measured by nitrogen physisorption analysis. In KOH-activated carbons, elevating the activation temperature resulted in a substantial enhancement of surface area, as it efficiently generated a porous network. In contrast, with $ZnCl_2$-activated carbons, the surface area diminished with increase in temperature. These findings strongly demonstrate the potential for PET waste as an effective feedstock for the synthesis of porous carbons, facilitating their application in energy storage and environmental remediation.

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Author contribution

Kendi Mwiti: Writing - original draft and editing, Conceptualization, Investigation, Data acquisition, Data analysis. *Bridget K. Mutuma*: Writing - Review and editing, Conceptualization, Supervision, Resources, Funding acquisition. *Martin Magu*: Writing - Review and editing, Supervision. *Dickson Andala*: Writing - Review and editing.

A. *Conflict of Interest*

The authors declare that they have no conflicts of interest related to the content of this manuscript.

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