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Program Studi Teknik Mesin, Fakultas Teknik, Universitas Lambung Mangkurat
Jl. Jenderal Achmad Yani KM 35,5 Banjarbaru, Kalimantan Selatan-70714 Telp-Fax : 0511-4773858
email: kinematika@ulm.ac.id

SIFAT FISIK DAN MORFOLOGI KOMPOSIT KARBON AKTIF TONGKOL JAGUNG

PHYSICAL AND MORPHOLOGICAL PROPERTIES OF CORN COB ACTIVATED CARBON COMPOSITE

Maulana Sanwijaya[1], Afira Ainur Rosidah[2], Muhammad Yunus[3]
[1,2]Mechanical Engineering, Institut Teknologi Adhi Tama Surabaya, Surabaya
[3]Research Center for Polymer Technology, National Research and Innovation Agency, Indonesia
email: maulanasanwijaya@gmail.com[1], afiraar@itats.ac.id[2]*, muha221@brin.go.id[3]

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Abstrak
Tongkol jagung termasuk ke dalam limbah pertanian yang kurang termanfaatkan secara optimal. Limbah organik ini merupakan salah satu kandidat yang tepat untuk dijadikan karbon aktif dengan berbagai kegunaan yang menjanjikan. Hal tersebut yang melatarbelakangi penelitian ini untuk membuat karbon aktif dari tongkol jagung yang diaktivasi menggunakan $ZnCl_2$ dengan variasi activator 0, 8, dan 16%. Pengujian FTIR dilakukan untuk mengonfirmasi gugus fungsi yang muncul pada karbon dari tongkol jagung sebelum dan sesudah aktivasi. Lalu karbon aktif yang telah dibuat kemudian dicampur tepung sagu dengan perbandingan 75:25 untuk menjadi komposit dan agar lebih mudah dalam fabrikasi sampelnya. Kemudian dilakukan karakterisasi SEM-EDX, kadar air, dan kadar abu. Hasil uji FTIR didapatkan bahwa penambahan aktivator $ZnCl_2$ menyebabkan munculnya gugus hidroksil. Pada uji kadar air mendapatkan hasil bahwa semakin tinggi konsentrasi aktivator maka akan semakin tinggi kadar airnya. Sedangkan semakin tinggi aktivator yang digunakan pada karbon aktif, kadar abu semakin rendah. Pengujian SEM menunjukkan perluasan ukuran dan keseragaman pori pada peningkatan konsentrasi $ZnCl_2$ dan hasil EDX menunjukkan peningkatan persentase unsur O.
Kata Kunci: tongkol jagung, karbon aktif, sifat fisik, morfologi

Abstract
Corn cobs are a type of agricultural waste that is not utilized optimally. This organic waste has potential as a raw material for producing activated carbon, which has various promising applications. This research aims to produce activated carbon from corn cobs, using $ZnCl_2$ as an activating agent with concentrations of 0%, 8%, and 16%. FTIR analysis was conducted to identify the functional groups present on the corn cob-derived carbon before and after activation. Additionally, the activated carbon produced was mixed with sago starch in a 75:25 ratio to create a composite, facilitating sample fabrication. SEM-EDX characterization, as well as water and ash content analyses, were subsequently performed. The FTIR results indicated that the addition of $ZnCl_2$ led to the appearance of hydroxyl groups. In terms of water content, results

showed that higher activator concentrations correlated with higher water content. Conversely, increasing the activator concentration in the activated carbon was associated with a decrease in ash content. SEM analysis revealed increased pore size and uniformity with higher $ZnCl_2$ concentrations, while EDX results demonstrated an increase in the percentage of oxygen elements.

Keywords: *corn cobs, activated carbon, physical properties, morphology*

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INTRODUCTION

Indonesia is an agricultural country with various high-quality commodities, including corn. The government continues to increase corn yields, which is the second staple food in Indonesia after rice, because corn is very important for human and animal food. Indonesia's annual harvest reaches 19,612,435 tons of corn which results in increased waste[1]. This waste is defined as organic waste. Organic waste is a type of waste made from organic materials such as food scraps, leaves, fruit peels, wood and so on[2,3]. Various efforts have been made to utilize corn cob waste that has not been optimized. Most corn cobs are only used as animal feed and burned to remove waste[4].

Previous studies show that corn cob waste is a promising source of carbon. Corn cobs can be converted into biochar and ground into carbon powder to reduce vehicle emissions[4]. Additionally, this carbon can be activated to produce a porous material, known as activated carbon, which can adsorb heavy metals and volatile organic compounds[5]. Activated carbon can be produced through physio-chemical activation, which involves both physical and chemical activation processes. Physical activation enlarges the pores by heating the material within a specific temperature range and holding time[6]. Chemical activation, on the other hand, utilizes reactive agents, such as alkaline (e.g., NaOH and Na_2CO_3), acidic (e.g., H_3PO_4 and H_2SO_4), and neutral/salt (e.g., $ZnCl_2$ and KCl) compounds[7-10]. Among these, $ZnCl_2$ is one of the most widely used chemical activators[11], [12]. However, despite its potential, there is limited research exploring the combined effects of physical and chemical activation using $ZnCl_2$ on the characteristics of activated carbon derived specifically from corn cobs, making this study a novel approach to optimizing corn cob-based activated carbon for adsorption applications.

The powdered form of activated carbon presents challenges in fabricating solid samples for certain types of testing. To address this, activated carbon is often combined with adhesive materials to enhance structure and shape retention, creating a composite. Composites are formed by combining two or more materials to achieve improved properties that differ from those of the individual components[13]. Activated carbon can be combined with sago starch, tapioca starch, or oil-based adhesives to form a solid structure[4], [14], [15]. Starch is a preferred adhesive due to its organic nature, low cost, and ease of availability. Recent studies have demonstrated that sago starch outperforms tapioca starch as a binder, yielding composites with higher calorific value and lower water and ash content[16,17]. Ningsih and Hajar reported that sago starch reduced water and ash content to 3.12% and 3.25%, respectively[17]. Nevertheless, there is limited research specifically investigating sago starch as an adhesive for activated carbon derived from corn cobs, making this study a novel approach to developing efficient, cost-effective composites for adsorption applications. In this research, sago starch was applied as the adhesive to produce a robust, practical composite from corn cob-derived activated carbon.

Since corn cobs are potential material to be synthesized as activated carbon and there is still some research gap in developing its properties, this research is concerned with exploring the morphology and physical properties of corn cob activated carbon composite. The carbon was activated using $ZnCl_2$ in the variation of 0, 8, and 16%, then characterized using Fourier Transform Infra-Red (FTIR). After the activation, the activated carbon is mixed to be a composite with the sago starch. Furthermore, the Scanning Electron Microscope-Energy Dispersive X-ray (SEM-EDX), water, and ash content were performed to represent the morphology and physical properties.

METHODS

The corn cobs (*Zea mays*) were collected from Blitar, East Java, Indonesia. The received corn cobs were dried for 3 days directly under the sun to reduce the water content. After drying, the corn cobs were burned using a pyrolysis technique for 30 minutes in a closed furnace to make the corn cobs into charcoal. Furthermore, the charcoal was crushed using a mortar and pestle to turn the charcoal into powder. Then the powder was sieved in 30 mesh to homogenize the particle size.

The activation process was performed in two steps. The first step is chemical activation using a $ZnCl_2$ solution. The $ZnCl_2$ solution varied in 0%, 8%, and 16% concentration. This activation was carried out by soaking the carbon powder for 5 hours in a beaker glass and then rinsing it using distilled water to neutralize the pH. After neutralizing the pH, the carbon was dried using oven for ±2 – 4 hours at 150°C. The second activation step is physical activation to enlarge the pores of the activated carbon. This physical activation was done inside a furnace at 300°C for 3 hours[6]. After the activation, carbon in each solution variation was analyzed using Fourier Transform Infra-Red (FTIR) to confirm the functional groups that appear. The FTIR method used Attenuated Total Reflectance (ATR) with spreading the sample directly to analyze.

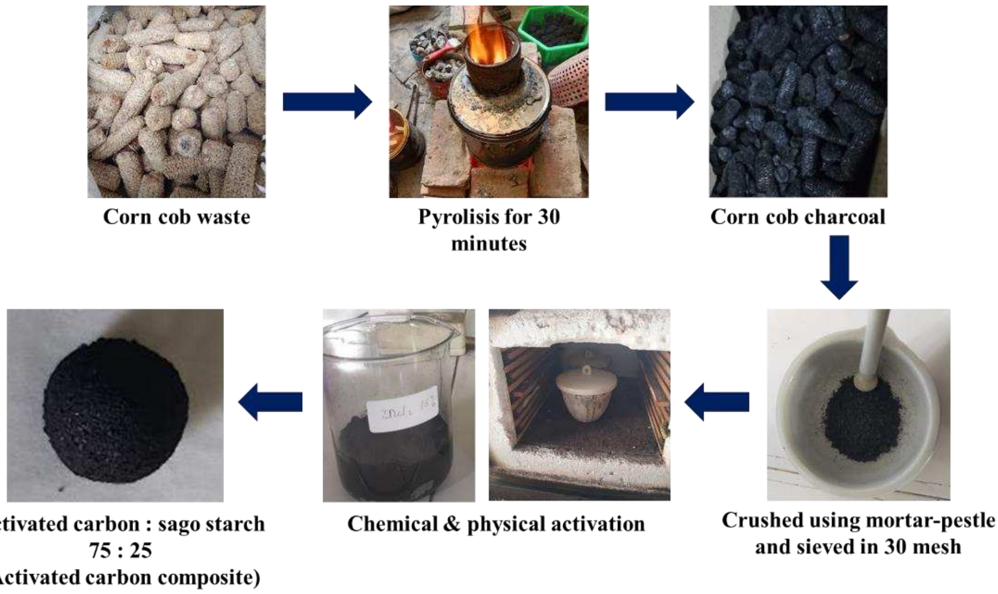


Figure 1. Corn cob activated carbon composite fabrication process

The activated carbon was mixed with sago starch in a composition of 75:25. Before mixing, 10 ml of the boiled water was poured into the sago starch first to make a sticky texture. Then, it was mixed with the activated carbon and shaped round like a briquette. This round shape is called activated carbon composite. The overall composite fabrication process is illustrated in Figure 1. Furthermore, the samples were performed water, ash content, and scanning electron microscopy with energy dispersive X-ray spectroscopy (SEM-EDX). The

SEM-EDX was used to analyze the morphology differenced and the elements observed before and after activation. The water content was performed in an oven at 110ºC until reached a constant mass. The water content was calculated by dividing the difference between the mass before and after heating by the mass before heating multiplied by 100%. After water content measurement, the specimens were performed ash content in a furnace at 550ºC for 3-4 hours. The ash content was measured with a similar method as the water content by dividing the mass after heating with the initial mass before heating inside the furnace[18].

RESULTS AND DISCUSSION

Fourier transform infra-red (FTIR) results

Figure 2 demonstrates the FTIR spectra of all corn cob carbon adsorbents after the activation process. The FTIR was performed in the range of 4000-500 cm^{-1}. The typical curves of the carbon with the addition of the $ZnCl_2$ exhibit different adsorption bonds. The corn cob activated carbon with 0% $ZnCl_2$ addition (**black line**) shows a typical activated carbon with C=C bond at the 1683 cm^{-1} wavenumber. This C=C bond also appears in the activated carbon with 8% (**red line**) and 16% (**blue line**) $ZnCl_2$ addition at a similar range of wavenumber.

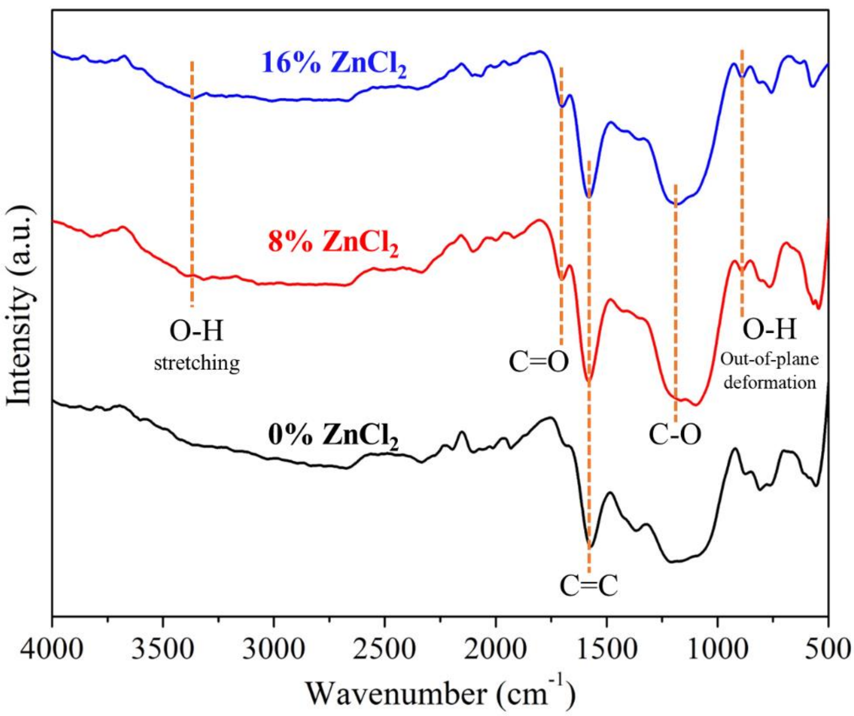


Figure 2. FTIR results of different $ZnCl_2$ concentration in corn cob carbon

The addition of 8% and 6% $ZnCl_2$ shows some different peaks that imply new adsorption bonds. The hydroxyl groups (O-H) are observed around the wavenumber of 3362 and 890 cm^{-1}. Furthermore, the ester groups (C-O bonds) appeared around the wavenumber of 1178 cm^{-1}. Another typical adsorption bond observed is C=O stretching around 1703 cm^{-1} wavenumber. These typical bonds are similarly founded in previous studies[19,20]. These results imply differences of the bonds appeared for each sample without and with activation. Moreover, the appearances of the oxygen-based bonds are further confirmed in the EDX analysis section.

Water content results

The water content was aimed to evaluate the percentage of water contained in the composite. The highest water content is reached by the value of 0.465% at the composite using activated carbon with 16% $ZnCl_2$ concentration. The lowest water content 0.394% with the composite using activated carbon with 0% $ZnCl_2$ concentration.

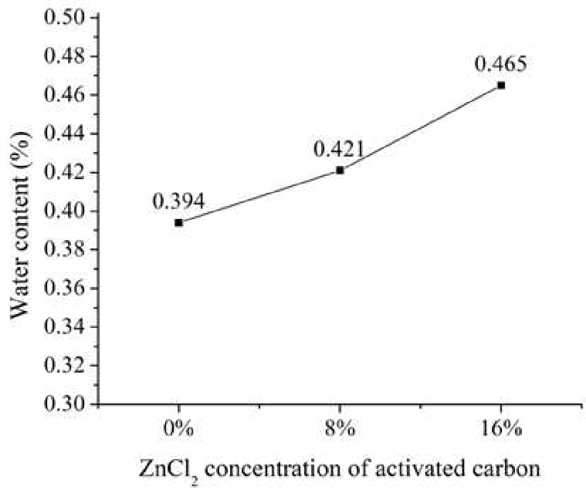


Figure 3. Water content results with increasing activator concentration

The water content of activated carbon increases with higher the activator ($ZnCl_2$) concentration, as illustrated in Figure 3. However, the rate of increase diminishes at higher concentration, with only a slight difference of approximately 0.02% between the samples activated with 8% and 16% of $ZnCl_2$ concentration. These findings align with previous studies which demonstrate a positive correlation between activation concentration and water content. This phenomenon can be attributed to the chemical activation process, wherein $ZnCl_2$ enhances the formation of micropores in the carbon structure. The activator ($ZnCl_2$) retains water molecules within these newly formed pores, particularly from the residual moisture introduced during the carbon washing stage. Consequently, the observed increase in water content reflects the extent of pore development facilitated by $ZnCl_2$ activation[21].

Ash content results

The ash content was analyzed to evaluate the residue or non-carbon content in the composite. Figure 4 shows that the highest ash content 0.379% is found in the composite using activated carbon with 0% $ZnCl_2$ concentration. Meanwhile, the lowest value 0.348% is achieved by the $ZnCl_2$ concentration of 16%.

The greater the concentration of $ZnCl_2$ activator in the carbon content, the lower the ash content. This trend is contrary to the result of water content. This result is similar to previous research, the higher the concentration of the activator, the lower the ash content produced. The decrease in ash content is due to the gas diffusion process in the carbon during the activation process, which is able to push out the remaining activator that still covers the carbon pores[21].

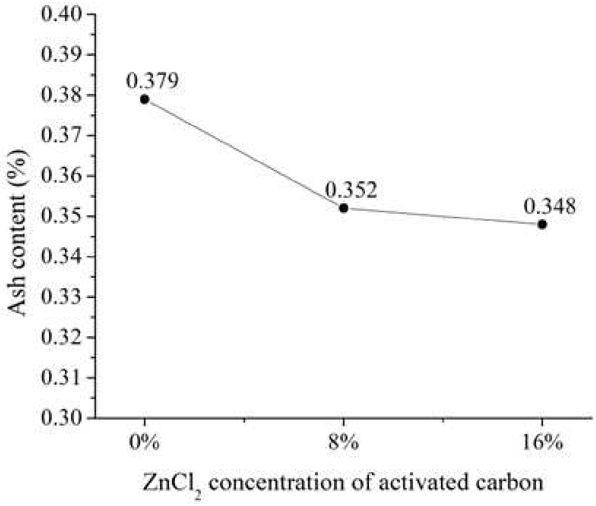
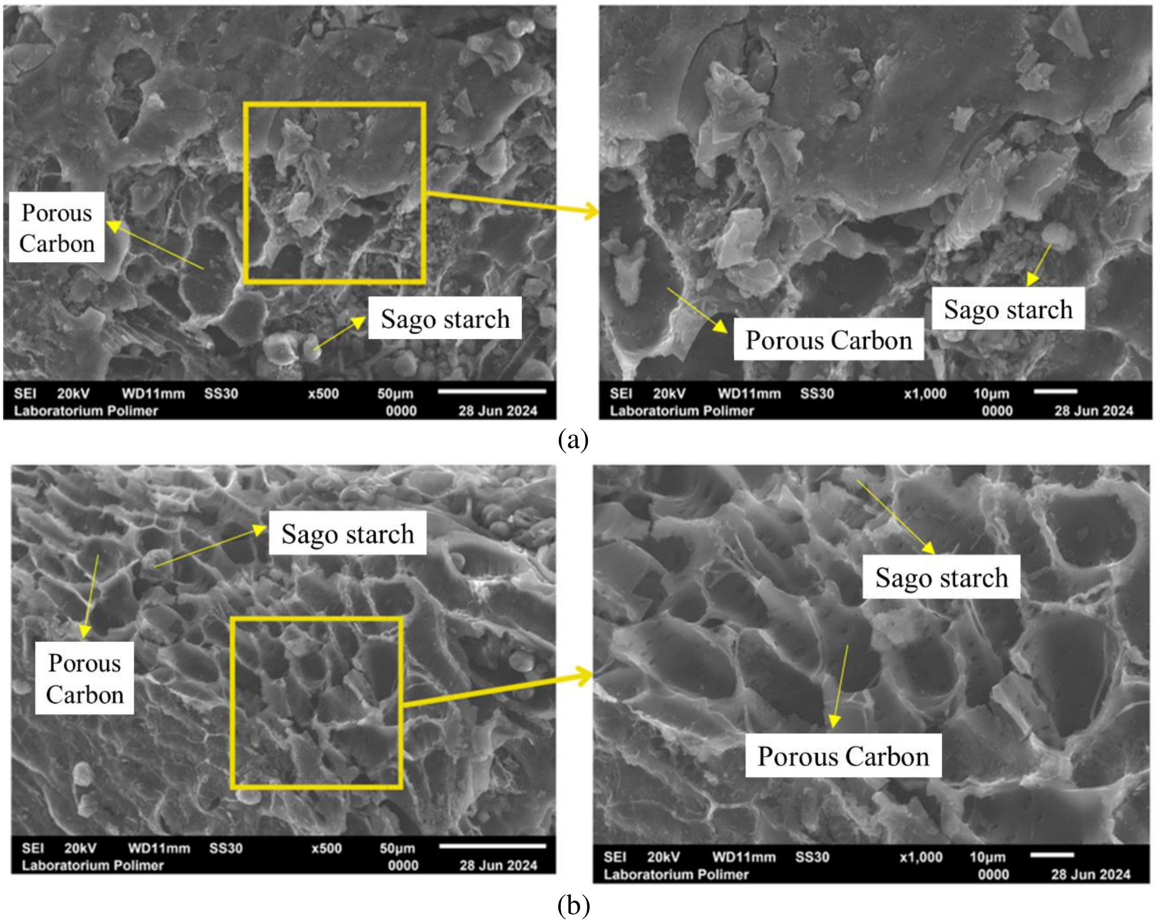
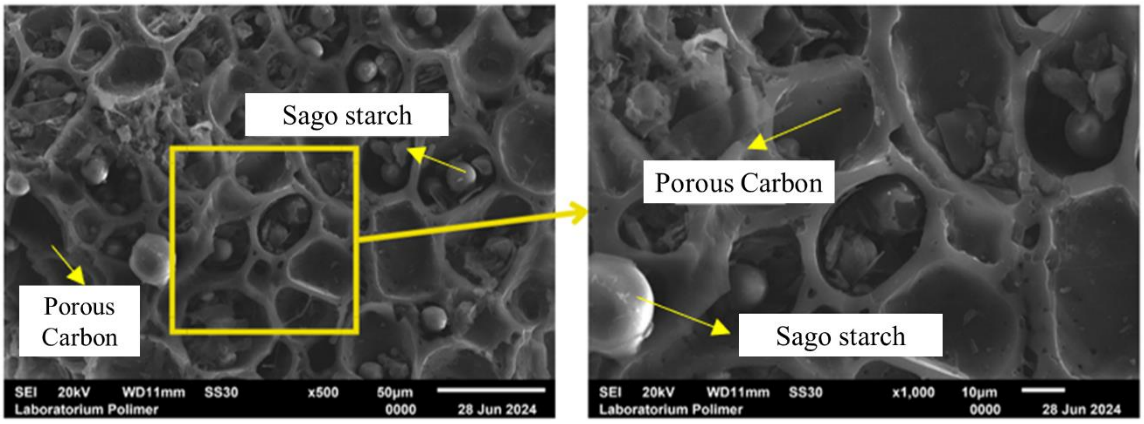


Figure 4. Ash content results with increasing activator concentration

Scanning electron microscopy with energy dispersive X-Ray spectroscopy (SEM-EDX) results

The SEM analysis was carried out to evaluate the morphological differences of the composite with or without activator. Moreover, the EDX was performed along with SEM to analyze the percentage of each element that appeared.





(c)

Figure 5. SEM image results of corn cob activated carbon composite (a) without activator (0%), (b) 8%, and (c) 16% $ZnCl_2$

In the SEM test results Figure 5 for 500x magnification on the left and enlarged at 1000x magnification on the right. The results show that without adding $ZnCl_2$ activator (0% $ZnCl_2$), the corn cob carbon composite exhibits a few pores in irregular form (Figure 5a). The addition of 8% $ZnCl_2$ increases the number of pores with more regular form. However, there are some irregular shapes and damage to the pore walls (Figure 5b). The highest addition 16% of $ZnCl_2$ indicates an outstanding morphology. The pore size is larger, more regular and complex without damage to its walls (Figure 5c).

The addition of activator affects the pore size and shape on the surface of activated carbon. In the SEM image results, the carbon is shown by a larger shape (in the form of pores) and for sago starch which is smaller (granules). The enlargement and uniformity of these pores are in accordance with research conducted by Amiruddin et al. [22]. These changes are possibly due to the physical activation that drives the volatile compounds to pass through the carbon surface and form pores. In addition, the chemical reaction between carbon atoms and $ZnCl_2$ plays a role in increasing the interlayer distance of carbon which possibly leads to the enlargement of the pore structure. The activation induces the formation of an aromatic structure[23] which is confirmed in the FTIR results and further proven in the EDX results. Based on Table 3, it can be inferred that the difference in concentration of the activator affects the elements that appeared in the composite such as the percentage of carbon (C), phosphor (P), and potassium (K) elements which decrease after the activation, but the oxygen (O) element increases. The increase in the O element is due to the formation of the aromatic structure in the activated carbon as confirmed in the FTIR result. Moreover, the loss of silicon (Si) elements in the composite, after the carbon was activated, is possibly due to the reaction with the $ZnCl_2$ that binds the silicon and wipes away at the rinsing process.

Table 3. The EDX results of the corn cob activated carbon composite

$ZnCl_2$ concentration of activated carbon	**Elements (%)**				
	C	**O**	**Si**	**P**	**K**
0%	84.03	14.64	0.32	0.32	0.69
8%	80.73	18.72	0	0.11	0.25
16%	80	18.88	0	0	0.12

CONCLUSION

The corn cob-based activated carbon was successfully synthesized and fabricated into a composite material. The FTIR analysis revealed characteristic adsorption peaks corresponding to O-H, C-O, and C=O bonds, which were intensified by the incorporation of $ZnCl_2$. This finding was further corroborated by the increased oxygen content detected in the EDX analysis. Water content analysis indicated that higher $ZnCl_2$ concentrations resulted in slightly increased water retention, likely due to $ZnCl_2$ occupying the carbon pores. Conversely, ash content decreased with higher $ZnCl_2$ concentrations, suggesting enhanced pore formation and a reduction in residual impurities. SEM-EDX analysis confirmed that $ZnCl_2$ activation improved pore size and structural uniformity, with increased oxygen content and a notable reduction in elements such as silicon and potassium. These results underscore the critical role of $ZnCl_2$ in modifying the structural and chemical properties of corn cob-based activated carbon, highlighting its potential for adsorption applications.

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