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Sewage Sludge-Derived Activated Carbon: Influence of Chemical Activation and Pyrolysis Conditions on Morphology and Crystallinity

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ABSTRACT

In this study activated carbon was prepared from sewage sludge through chemical activation using different chemicals such as (ZnCl2, NaOH, KOH) followed by pyrolysis in tubular furnace at (500°C, 600°C, 700°C) and further physically activated using CO2 at 400°C. Pure sludge and prepared ACs were analyzed using SEM, FTIR and XRD. The sample morphology in SEM shows that the surface morphology of each sample is quite different according to the used chemical activating reagents and relatively larger pores on the surface of the prepared activated carbon using NaOH solution for activation and pyrolysis at 500oC compared to other samples. The results showed 18% crystallinity compared to that of commercial AC with 44% crystallinity. The results of FT-IR demonstrate that the properties of the post-treated affect the final products and depend on the method used and that it contains similar functional groups to those present in the commercial AC. During carbonation process, the sludge sample lost 30-60 % of the original weight.

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INTRODUCTION

The increasing generation of sewage sludge as a byproduct of wastewater treatment plants presents a significant environmental challenge (Bian et al. 2018). Sewage sludge is rich in organic and inorganic matter, making it a potential resource for conversion into value-added materials, such as activated carbon (AC) (Yin, Liu & Ren 2019). Activated carbon has gained widespread application due to its high surface area, porous structure, and adsorption capabilities, making it an excellent candidate for environmental remediation, including water purification and air filtration (Björklund & Li 2017). Traditional production of activated carbon primarily relies on non-renewable sources like coal and wood, leading to increased interest in developing sustainable alternatives (Gan 2021). Sewage sludge, being a waste material, offers an eco-friendly and low-cost precursor for producing activated carbon while simultaneously addressing waste management issues (Montoya-Bautista, Mohamed & Li 2022).

The preparation of activated carbon involves two main processes: chemical or physical

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activation, followed by pyrolysis. Chemical activation typically involves treating the precursor with activating agents such as $ZnCl_2$, NaOH, or KOH, which help to develop porosity by removing volatiles and increasing the carbon content (Wang et al. 2021). The pyrolysis step, conducted at elevated temperatures, further enhances the material's physical and chemical properties, including its pore size, surface area, and structural stability (Zhang et al. 2023). However, the activation method and pyrolysis conditions significantly affect the morphology, surface chemistry, and crystallinity of the final activated carbon product (Almahbashi et al. 2021).

In particular, chemical activation can influence pore development and functional group formation on the carbon surface, while the pyrolysis temperature controls the extent of carbonization and the evolution of the carbon structure (Kumar et al. 2022). Research has shown that lower pyrolysis temperatures (around 500°C) typically result in higher surface areas and larger pore volumes due to incomplete carbonization, whereas higher temperatures (600–700°C) tend to enhance the crystallinity and structural integrity of the material (Bao et al. 2020). Furthermore, the type of chemical activator plays a critical role in determining the pore size distribution and adsorption properties of the activated carbon (Liu et al. 2022).

Despite growing interest in sewage sludge-derived activated carbon, the influence of different activation agents and pyrolysis conditions on its morphology and crystallinity is not yet fully understood (Aljubiri et al. 2024). Therefore, this study aims to investigate the effects of chemical activation using $ZnCl_2$, NaOH, and KOH, followed by pyrolysis at 500°C, 600°C, and 700°C, on the structural properties of the resulting activated carbon. The activated carbon samples were characterized using scanning electron microscopy (SEM), Fourier transform infrared spectroscopy (FTIR), and X-ray diffraction (XRD) to analyze their morphological and crystallographic features. By understanding how activation and pyrolysis conditions affect the material's performance, this research contributes to the development of more efficient sewage sludge-derived adsorbents for environmental applications.

MATERIALS & METHODS

Materials

The sewage sludge used in this study was collected from Nizwa Sewage treatment plant, Nizwa, Sultanate of Oman. The sludge was dewatered and dried at 105°C for 24 hours in a drying oven to remove excess moisture. Three chemical reagents, $ZnCl_2$ (zinc chloride), NaOH (sodium hydroxide), and KOH (potassium hydroxide), were used as chemical activators for the activation process. All the chemicals used in this were of analytical grade.

Chemical Activation of Sewage Sludge

Dried sewage sludge was chemically activated using three different chemicals: $ZnCl_2$, NaOH, and KOH. The activation was performed by mixing the sludge with each chemical activator in a 1:1 ratio by weight. The chemical sludge mixture was stirred thoroughly to ensure uniform distribution of the activating agent throughout the sludge matrix. The mixture was then left to stand at room temperature for 24 hours to allow for complete interaction between the sludge and the chemical activator.

Pyrolysis (Carbonization) Process

After chemical activation, the sludge samples were subjected to pyrolysis in a tubular furnace. The furnace was preheated to the desired temperatures of 500°C, 600°C, and 700°C to study the influence of temperature on the properties of the activated carbon. The samples were placed in a ceramic crucible and pyrolyzed under an inert nitrogen atmosphere at a flow rate of 100 mL/min to prevent oxidation. The samples were held at the final temperature for 1 hour

before being cooled to room temperature under continuous nitrogen flow. This process resulted in carbonized sludge with varying degrees of porosity and structure based on the temperature and chemical activator used.

Characterization Techniques

Scanning Electron Microscopy (SEM)

The surface morphology of the raw sewage sludge, as well as the chemically activated carbon samples, were analyzed using scanning electron microscopy (SEM) available at Daris laboratory at the University of Nizwa. Samples were sputter-coated with gold to enhance conductivity and imaged under different magnifications to observe the development of porosity and changes in surface texture. The SEM analysis will help to determine the pore size and distribution on the surface of the activated carbon samples.

Fourier Transform Infrared Spectroscopy (FTIR)

Fourier Transform Infrared Spectroscopy (FTIR) available at Daris laboratory, University of Nizwa was used to identify the functional groups present on the surface of both raw sludge and activated carbon samples. The spectra were recorded in the wavelength range of 4000–400 cm^{-1} using a KBr pellet method.

X-Ray Diffraction (XRD)

X-Ray Diffraction (XRD) analysis was conducted at University of Nizwa Daris lab to evaluate the crystallinity of the activated carbon samples. The XRD patterns were recorded using a diffractometer equipped with Cu-Kα radiation (λ = 1.5406 Å) at a scanning rate of 2°/min over a 2θ range of 10°–80°. The crystallinity of the activated carbon was compared to that of commercial activated carbon.

Mass Loss Measurement

The mass loss during the pyrolysis process was measured by weighing the sludge samples before and after pyrolysis. The weight loss was expressed as a percentage of the original mass.

Energy-dispersive X-ray spectroscopy (EDS)

Energy-dispersive X-ray spectroscopy was used to analyze the elemental composition of sewage sludge and activated carbon prepared from sewage sludge.

RESULTS AND DISCUSSION

Characterization of prepared activated carbon-based sewage sludge

SEM analysis

The surface morphology of sewage sludge-derived activated carbon was characterized using Scanning Electron Microscopy (SEM) to assess the influence of chemical activation agents and pyrolysis temperatures on structural characteristics, as these parameters are crucial for optimizing adsorption properties (Fachini & Figueiredo 2022; Raheem et al. 2018). The SEM images of produced carbons are presented in Fig. 1-4. All the SEM images give the evidence that all the carbons produced in this study possessed porous structure. Significant differences in surface topography, pore structure, and morphology were observed based on the choice of activating agent—$ZnCl_2$, NaOH, or KOH—and the pyrolysis temperatures of 500°C, 600°C, and 700°C, each affecting the pore distribution and size (Shi et al. 2021).

Effect of Activating Agents on Surface Morphology

$ZnCl_2$-Activated Carbon: SEM analysis of $ZnCl_2$-activated carbon displayed in Fig.1

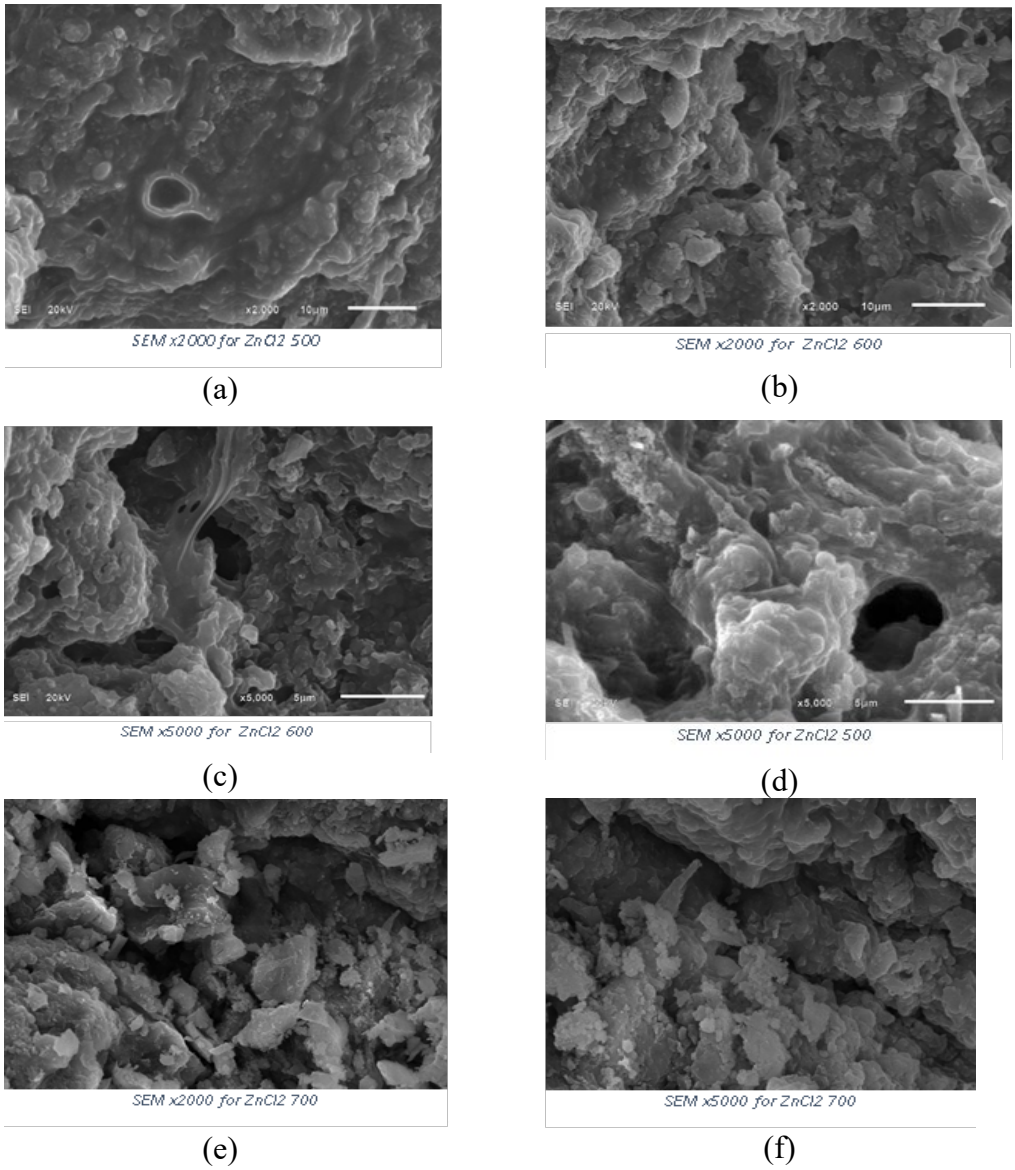


Fig. 1. SEM photographs of sewage sludge activated by ZnCl2

shows a compact, dense morphology with limited pore development, reflecting $ZnCl_2$'s milder dehydration effect during activation. $ZnCl_2$ primarily acts as a dehydrating agent, producing a denser structure with fewer and smaller pores. This limited porosity is attributed to $ZnCl_2$'s restricted gasification and minimal removal of organic components, resulting in a more compact structure and less surface area for adsorption (Shi et al. 2021; Raheem et al. 2018).

NaOH-Activated Carbon: As shown in Fig.2 SEM analysis of NaOH-activated carbon pyrolyzed at 500°C revealed a well-developed porous structure, with relatively larger pores compared to other chemically activated samples. This indicates that NaOH facilitates extensive pore formation during carbonization, likely due to its strong dehydrogenating and oxidizing properties, which enhance the removal of volatile components and break down organic matter, resulting in a more interconnected pore network (Raheem et al. 2018). The enhanced porosity and larger pore size provide an increased surface area, beneficial for applications in adsorption (Zhao et al. 2023; Reddy 2006).

KOH-Activated Carbon: The KOH-activated carbon showed in Fig.3 is more granular, less porous structure, with a combination of micro- and mesopores, suggesting that KOH

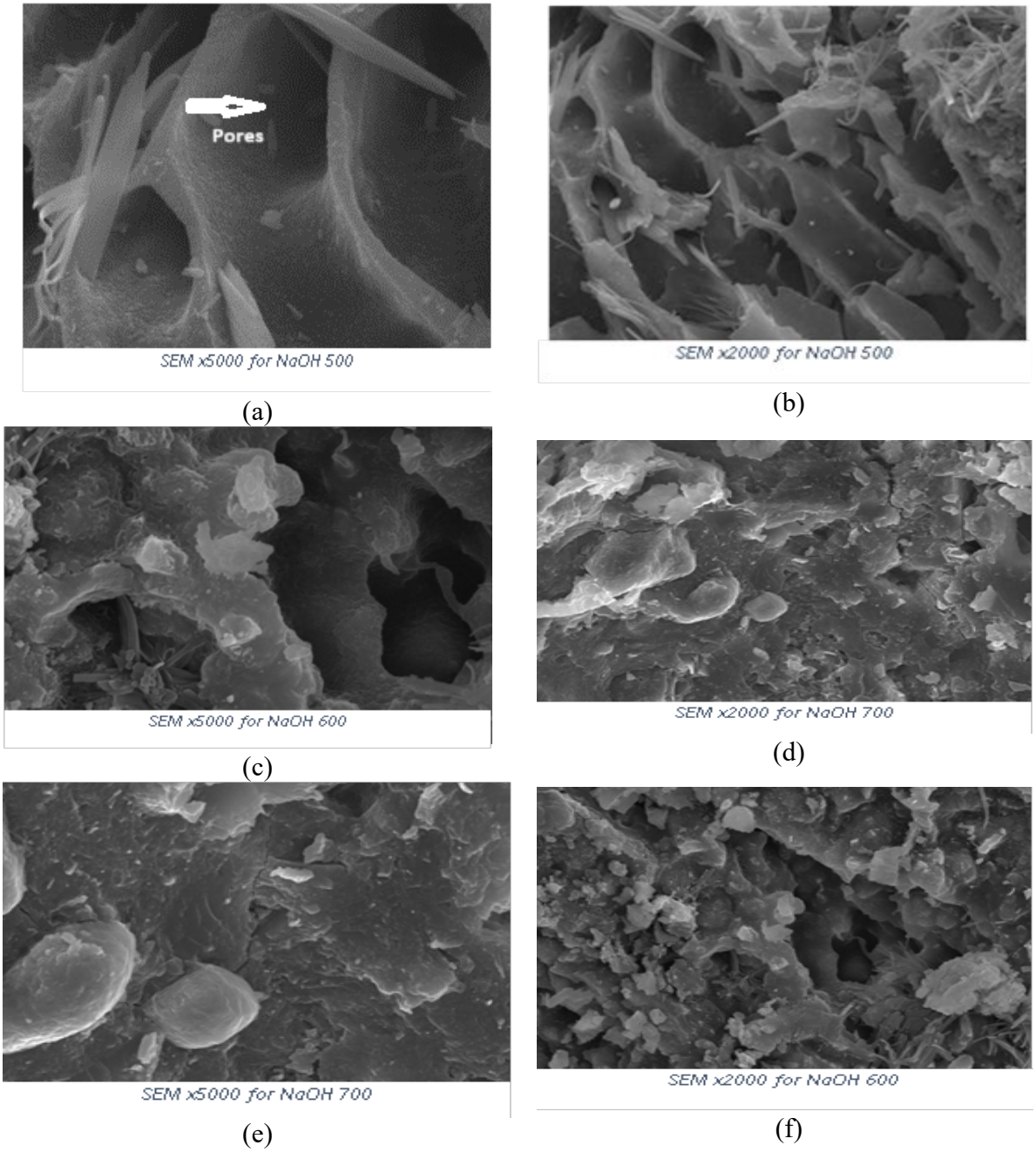


Fig. 2. SEM photographs of sewage sludge activated by NaOH

activation partially oxidizes the carbon matrix but results in less pronounced porosity than NaOH. The irregularities in surface morphology, as seen in the SEM images, indicate that KOH activation may induce partial carbon burn-off, leading to a more heterogeneous pore structure and contributing to its unique adsorption characteristics (Al Dawery et al. 2023).

Effect of Pyrolysis Temperature on Surface Morphology

The pyrolysis temperature significantly influenced the surface structure of the activated carbon, with higher temperatures (600°C and 700°C) causing pronounced changes, including reduced porosity and surface collapse (Zhao et al. 2023).

At 500°C samples activated with NaOH and pyrolyzed at 500°C, SEM images revealed

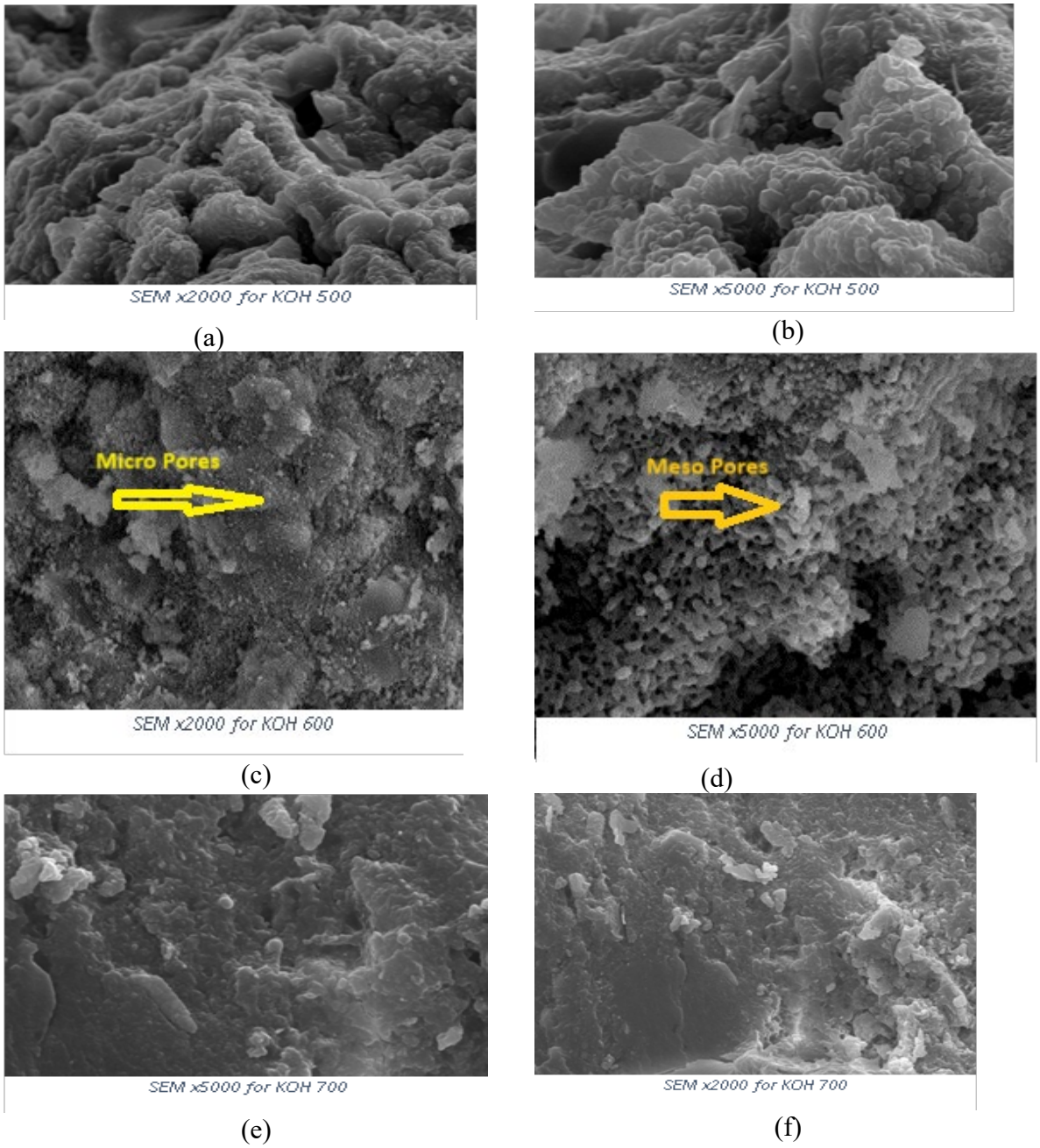


Fig. 3. SEM photographs of sewage sludge activated by KOH

a well-preserved pore structure with defined open pores, resulting in a highly porous matrix suitable for adsorption applications. The moderate temperature preserved the integrity of the carbon matrix, preventing collapse and allowing for the retention of porosity and surface area, which are critical for effective adsorption (Al Dawery et al. 2023). SEM images of samples pyrolyzed at 600°C and 700°C showed a smoother, less porous surface, with significant pore collapse due to the effects of higher thermal energy. These temperatures likely caused excessive burn-off, resulting in shrinkage of the carbon matrix and sealing of smaller pores, thereby reducing the overall porosity and surface area (Fachini & Figueiredo 2022). Excessive carbon burn-off at elevated temperatures led to a more compact structure, consistent with reduced adsorption capability (Zhao et al. 2023).

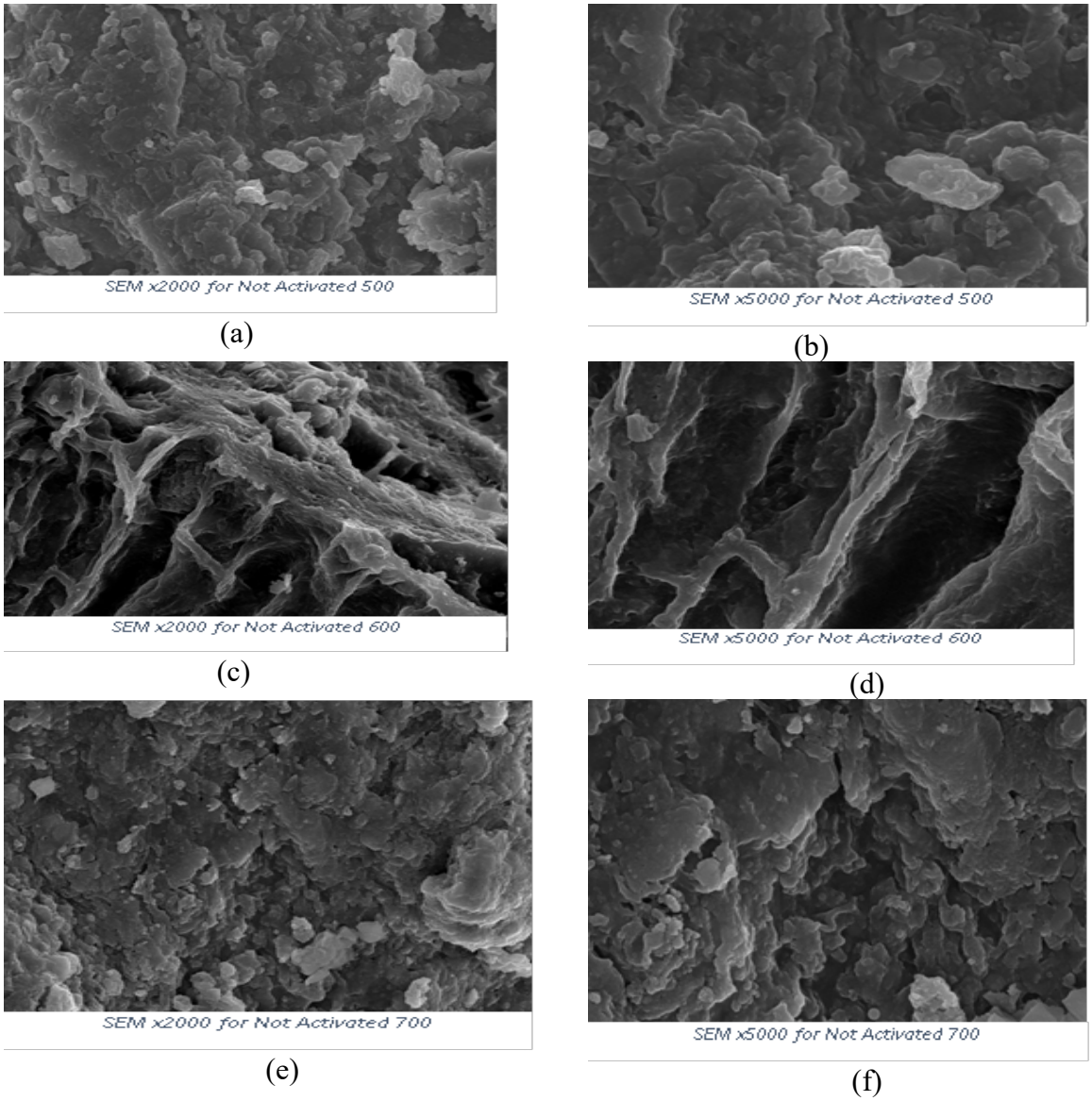


Fig. 4. SEM photographs of sewage sludge not activated

XRD analysis

X-ray diffraction (XRD) analysis was conducted to evaluate the crystallinity of the sewage sludge-derived activated carbon (AC) samples, both with and without chemical activation, and to compare them with commercial activated carbon. Results of XRD analysis ins presented in Table 1. Crystallinity is a key structural property that impacts the adsorption capacity of activated carbons by influencing their porosity, surface area, and stability.

Crystallinity of Commercial Activated Carbon

The XRD results shown in Table 1. indicated that the commercial activated carbon exhibited a crystallinity of 44.6%, with the remaining structure being amorphous. The high level of crystallinity in commercial AC is due to its well-ordered carbon lattice, which results from the controlled production processes it undergoes. This ordered arrangement enhances its surface stability and provides a robust framework for adsorption applications (Tian et al. 2020).

Table 1. XRD of prepared and commercial activated carbon.

Types of adsorbents	Crystallinity structures %	Amorphous structures%
Commercial AC	44.6	55.4
Pure sludge	15.2	84.8
Prepared AC without chemical activation at 500ºC	15.8	84.2
Prepared AC activated with $ZnCl_2$ at 500ºC	18.1	81.9
Prepared AC activated with NaOH at 500ºC	23.6	76.4
Prepared AC activated with KOH at 500ºC	17.1	82.9

Crystallinity of Pure Sludge-Derived Activated Carbon

The XRD analysis of the activated carbon derived from pure sewage sludge (without chemical activation) revealed a lower crystallinity of 15.2%. The reduced crystallinity can be attributed to the heterogeneity of the sludge precursor and the absence of strong chemical agents that promote structural rearrangement during pyrolysis. Sludge-derived activated carbon typically contains various inorganic impurities, leading to a less organized carbon structure and increased amorphous content. This contributes to its lower structural order and reduced crystallinity, which could limit its adsorption efficiency compared to commercial AC (Zhao & Zhang 2024).

Effect of Chemical Activation on Crystallinity

Chemical activation plays a significant role in altering the crystallinity of the prepared activated carbon. Among the different chemicals used ($ZnCl_2$, NaOH, KOH), the highest crystallinity was observed in the sample activated with NaOH, which exhibited a crystallinity of 23.6%. This increase in crystallinity is due to NaOH's ability to etch away non-crystalline components during activation, resulting in a more ordered structure. The NaOH activation also promotes the formation of a porous carbon matrix, which enhances both crystallinity and adsorption properties (Al Dawery et al. 2023).

NaOH-Activated Carbon

The NaOH-activated sample demonstrated the highest crystallinity among the prepared ACs, suggesting that NaOH is effective in producing a more ordered carbon structure. This increase in crystallinity can be linked to the removal of amorphous materials and the development of a more graphitic structure during the activation process. The carbonization temperature of 500°C also plays a role, as it is high enough to promote structural reorganization but not too high to cause excessive amorphization (Wang et al. 2021).

KOH and $ZnCl_2$-Activated Carbons

The activated carbon samples prepared using KOH and $ZnCl_2$ exhibited crystallinity levels between those of pure sludge and NaOH-activated samples. The chemical action of these activating agents is less aggressive than NaOH in terms of promoting crystallization. $ZnCl_2$ primarily acts as a dehydrating agent, resulting in a higher amorphous content, while KOH produces a moderate degree of crystallinity due to its relatively mild etching effect (Zhao & Zhang 2024).

Amorphous Nature of Sewage Sludge-Derived Activated Carbons

Despite the improvements in crystallinity with chemical activation, all sewage sludge-derived activated carbon samples retained a significant amorphous fraction, reflecting the inherent complexity of the sludge precursor. The amorphous nature is primarily due to the presence of non-carbon components such as silica, heavy metals, and other inorganic compounds**,

which inhibit the formation of a fully crystalline structure. These impurities, along with the heterogeneity of the sludge, contribute to the disordered arrangement of carbon atoms, resulting in a lower overall crystallinity compared to commercial AC (Wang et al. 2021).

The amorphous regions in the activated carbon are not necessarily a disadvantage, as they provide high surface areas and are responsible for the development of microporosity, which is crucial for adsorption. Therefore, while higher crystallinity improves structural stability, a balance between crystalline and amorphous phases is desirable for optimizing the adsorption properties of activated carbons (Wang et al. 2021).

Crystallinity and Pyrolysis Conditions

The crystallinity of the prepared activated carbon samples was also influenced by the pyrolysis conditions. The pyrolysis temperature of 500°C was sufficient to promote some degree of crystallization, especially in the NaOH-activated samples, but higher temperatures (600°C and 700°C) might further increase crystallinity at the cost of reducing porosity. The physical activation using CO_2 at 400°C further contributed to the removal of amorphous carbon, though its impact on crystallinity was less pronounced compared to chemical activation (Tian et al. 2020).

Comparison of Crystallinity: Prepared ACs vs. Commercial AC

As shown in Table 1. compared to the 44.6% crystallinity of commercial activated carbon, the sewage sludge-derived ACs exhibited lower crystallinity across all samples. However, the NaOH-activated carbon showed the most significant improvement, achieving 23.6% crystallinity, which is still well below that of commercial AC but represents a substantial increase over pure sludge-derived AC. This difference is due to the industrial-grade processing of commercial AC, which includes highly controlled activation and carbonization conditions to optimize the crystallinity and adsorption properties of the material (Zhao & Zhang 2024).

FTIR analysis

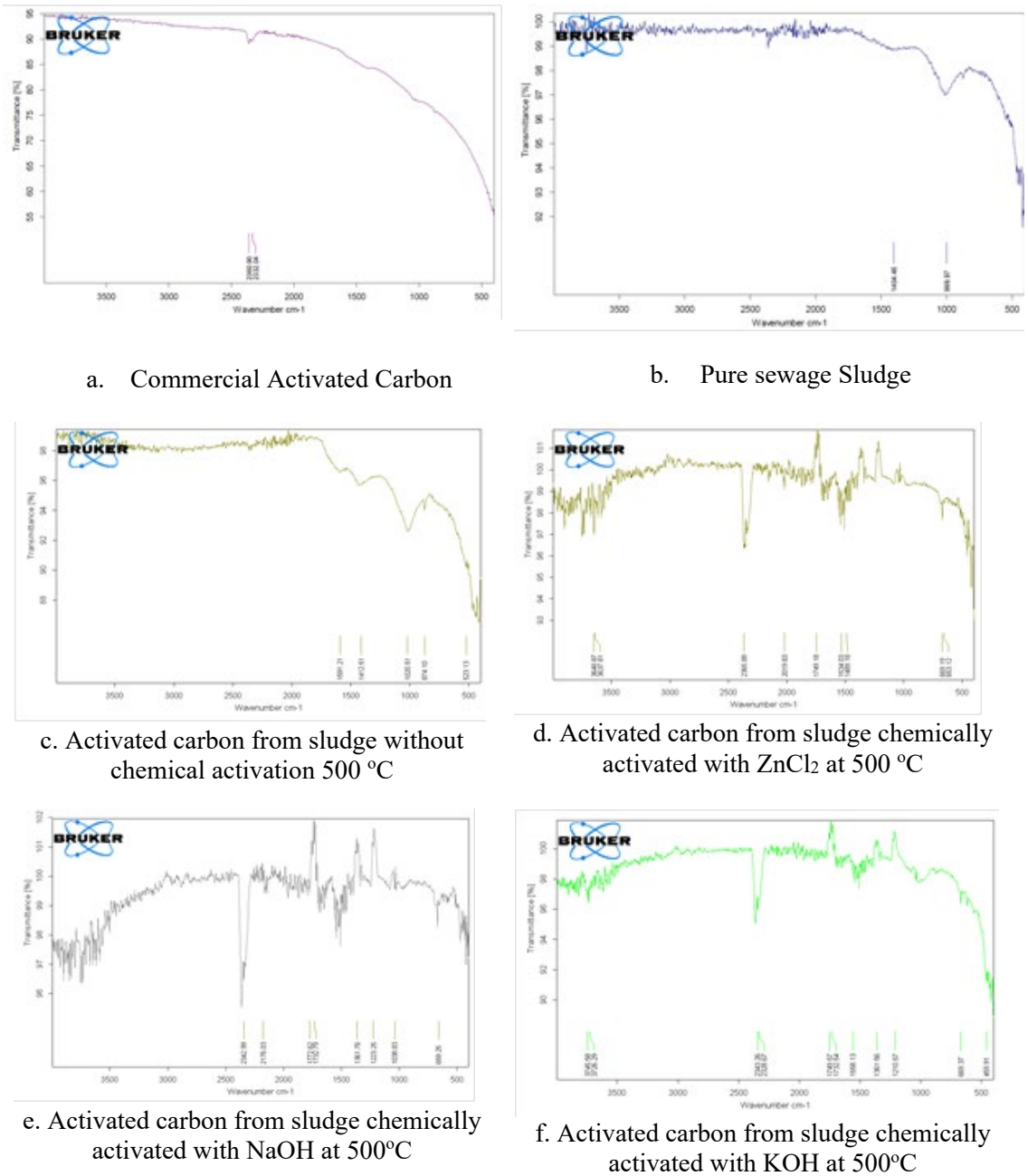
Fourier Transform Infrared Spectroscopy (FTIR) was employed to identify the functional groups present in the activated carbon (AC) samples derived from sewage sludge. The FTIR spectra shown in Fig.5a to 5f were analyzed for both chemically activated and non-activated (pure) sludge samples and compared with commercial activated carbon. This analysis provides insights into the surface chemistry of the prepared ACs, which plays a crucial role in their adsorption capabilities.

Functional Groups in Commercial Activated Carbon and Pure Sludge

The FTIR spectrum of the commercial activated carbon shown in Fig.5a & 5b exhibited two characteristic absorption bands at 2360 cm^{-1} and 2332 cm^{-1}, corresponding to the presence of the O=C=O group (carbon dioxide). These bands are typically associated with CO_2 adsorbed on the surface or possibly carboxyl groups. The pure sludge sample, on the other hand, showed fewer defined peaks, indicating a lack of well-developed surface functional groups compared to activated carbon. This underscores the role of chemical activation and pyrolysis in enhancing the surface chemistry of sludge to form more functionalized activated carbon (Cao et al. 2024; Fachini & Figueiredo 2022).

Effect of Chemical Activation on Functional Group Development

All chemically activated carbon samples ($ZnCl_2$, NaOH, KOH) exhibited absorption patterns similar to commercial activated carbon, particularly in the range of 2300-2350 cm^{-1}, suggesting the presence of the CO_2 functional group. This band is likely due to the physical activation using CO_2 gas during the final stage of activation, which introduced carboxyl or carbonate



a. Commercial Activated Carbon

b. Pure sewage Sludge

c. Activated carbon from sludge without chemical activation 500 ºC

d. Activated carbon from sludge chemically activated with $ZnCl_2$ at 500 ºC

e. Activated carbon from sludge chemically activated with NaOH at 500ºC

f. Activated carbon from sludge chemically activated with KOH at 500ºC

Fig. 5. FTIR analysis of prepared, commercial activated carbons and pure sludge

groups on the surface of the activated carbon (Boualem et al. 2014).

NaOH-Activated Carbon

The NaOH-activated sample showed distinct peaks in the FTIR spectrum, indicating the introduction of a variety of oxygen-containing functional groups, which are known to improve adsorption properties. For instance, a strong absorption band in the range of 1730-1780 cm^{-1} corresponds to C=O stretching, commonly found in aromatic carbonyl groups. This suggests that NaOH activation promotes the formation of carboxyl and ketone groups, which can enhance the binding of pollutants through electrostatic interactions and hydrogen bonding (Cao et al. 2024).

KOH-Activated Carbon

Similar to NaOH, the KOH-activated carbon displayed absorption in the 1730-1780 cm^{-1} region, indicating the presence of aromatic C=O groups. Additionally, KOH activation produced peaks in the region 1210-1370 cm^{-1}, corresponding to C-N stretching. This suggests that nitrogen-containing functional groups may be introduced during KOH activation, contributing to the adsorption of metal ions and organic pollutants (Boualem et al. 2014).

$ZnCl_2$-Activated Carbon

The $ZnCl_2$-activated carbon showed less pronounced functional group diversity compared to NaOH and KOH activation. However, the FTIR spectrum still exhibited significant absorption in the C=O stretching region and the CO_2-related bands. $ZnCl_2$, acting as a dehydrating agent, is more effective at producing a carbon-rich surface but less effective at introducing oxygenated functional groups (Cao et al. 2024).

Aromatic Functional Groups and Carbon Backbone

The FTIR spectra of the chemically activated samples also revealed peaks around 990-1000 cm^{-1}, corresponding to C=C bending. This band is associated with the presence of aromatic rings, indicating that the carbonization process retains some of the aromatic structures present in the original sludge. The presence of these aromatic C=C bonds highlight the transition of sewage sludge into a carbonized material with a graphitic backbone, which is crucial for maintaining structural integrity and providing a high surface area for adsorption (Boualem et al. 2014).

Comparison with Commercial Activated Carbon

While the FTIR spectra of the chemically activated sewage sludge-derived ACs showed similarities to commercial activated carbon, the intensity of the absorption bands varied. The commercial activated carbon showed stronger absorption in the O=C=O region, indicating a higher concentration of carboxyl or carbonate groups. However, the chemically activated sludge-derived ACs, particularly those treated with NaOH, exhibited additional functional groups such as C=O and C-N groups, which may provide a wider range of interactions for adsorbing different types of pollutants (Blachnio et al. 2020).

Role of Activation Method in Functional Group Formation

The activation method significantly influenced the type and concentration of functional groups on the surface of the prepared ACs. NaOH-activated carbon demonstrated the most diverse range of functional groups, which can be attributed to the strong basic nature of NaOH, promoting the formation of oxygen-containing and nitrogen-containing groups during activation. In contrast, $ZnCl_2$ activation, which primarily acts as a dehydrating agent, resulted in fewer functional groups, producing a carbon material with less surface reactivity (Blachnio et al. 2020).

The introduction of CO_2 activation also contributed to the formation of carbonate-like structures on the surface of the ACs, as indicated by the bands in the 2300-2350 cm^{-1} region. This treatment likely enhanced the porosity of the material while introducing additional functional groups, making the activated carbon more effective for pollutant removal (Almahbashi et al. 2021).

Weight Loss and Functional Group Correlation

During the carbonization process, the weight loss of sludge samples ranged between 30% and 60%, depending on the temperature and the chemical used for activation, as depicted in Fig 6. A weight loss nearing 60% was observed for most samples carbonized at 700°C, except for

those activated with $ZnCl_2$. This exception may be attributed to $ZnCl_2$'s contribution to stronger intermolecular forces, resulting in a more robust bond between sludge particles. Additionally, the EDS results showed that carbon content decreased across all pyrolysis temperatures, with $ZnCl_2$-activated samples exhibiting the least reduction (Table 2). This suggests that $ZnCl_2$ has a unique influence on the material, possibly due to its lower reactivity with the mineral and carbonate components in the sludge, which would otherwise generate CO_2 and water. The chemical reagents used in activation may also react with organic species, leading to the formation of cross-linked biopolymer fragments.

The observed weight loss (30-60%) during carbonization was accompanied by the removal of volatile organic compounds, contributing to the development of new functional groups. FTIR analysis confirmed the formation of carboxyl, carbonyl, and carbonate groups on

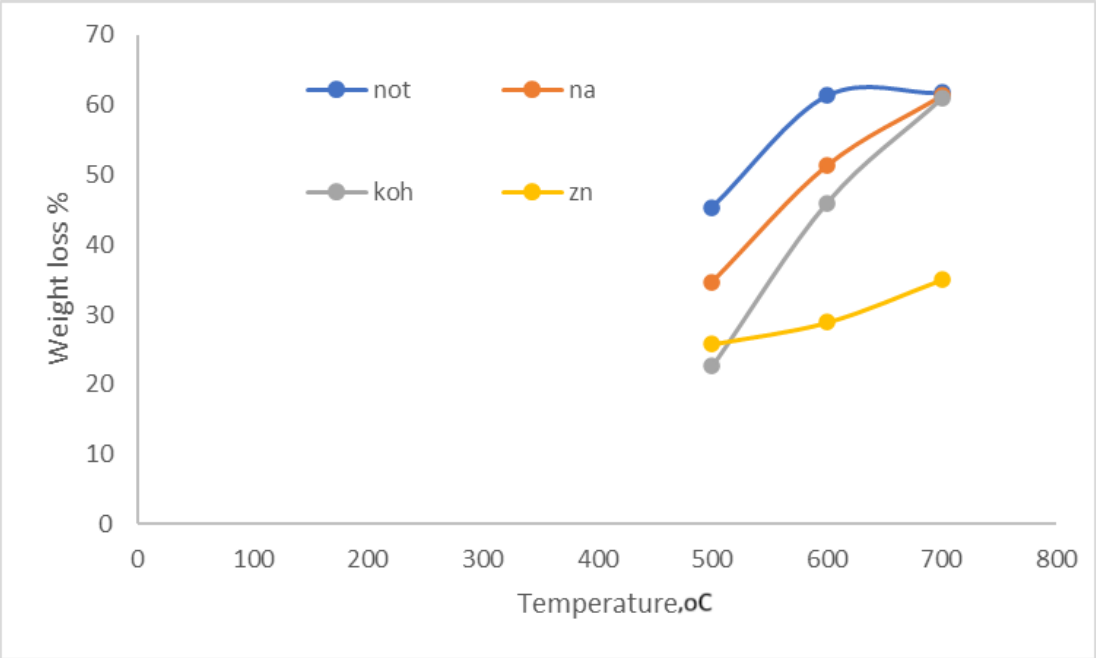


Fig. 6. Weight loss during carbonization process

Table 2. Percentage of carbon changing during carbonization process.

Activation agent	Pyrolysis temperature °C	% carbon content	Si%	O%
Original sample	No pyrolysis	32.03	4.15	8.21
$ZnCl_2$	500	17.24	5.2	5.3
$ZnCl_2$	600	12.69	4.1	4.89
$ZnCl_2$	700	7.82	4	4.16
NaOH	500	16.56	6.6	11.56
NaOH	600	9.97	6.6	6.7
NaOH	700	6.2	6.2	7.6
KOH	500	18.33	6	6.46
KOH	600	16.70	4.5	4.5
KOH	700	3.66	4.67	6.2
Not chemically activated	500	25.75	4.32	5.8
Not chemically activated	600	10.94	6	4.47
Not chemically activated	700	9.31	4.8	5.76

the surface of the material, with NaOH and KOH activation resulting in more pronounced functionalization compared to $ZnCl_2$ (Almahbashi et al. 2021). SEM analysis further supported these findings, revealing that the restructuring of the carbon matrix and the removal of volatile organic compounds led to a more porous and less dense material. The weight loss, particularly significant in NaOH-activated samples, correlated with extensive pore formation, demonstrating that chemical activation followed by controlled pyrolysis is an effective method for producing highly porous activated carbon.

CONCLUSIONS

In conclusion, this study demonstrates the effectiveness of chemical activation, especially using NaOH, in enhancing the crystallinity, porosity, and surface chemistry of sewage sludge-derived activated carbon (AC). Although NaOH-activated AC exhibits lower crystallinity compared to commercial AC, its high amorphous content and well-developed microporosity make it highly suitable for adsorption applications. The introduction of functional groups such as C=O and C-N, confirmed by FTIR analysis, further enhances its adsorption capacity, while SEM analysis reveals a favorable porous structure, particularly in NaOH-activated samples pyrolyzed at 500°C. $ZnCl_2$ activation resulted in a more compact and less porous material, while KOH activation provided intermediate results.

Overall, the findings underscore that NaOH activation, combined with controlled pyrolysis, produces AC with superior surface properties and high adsorption potential. Despite lower crystallinity, sludge-derived ACs offer a balance of high surface area, porosity, and functional group diversity, making them competitive with commercial ACs for various environmental and industrial adsorption applications.

GRANT SUPPORT DETAILS

The present research did not receive any financial support.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interest regarding the publication of this manuscript. In addition, the ethical issues, including plagiarism, informed consent, misconduct, data fabrication and/ or falsification, double publication and/or submission, and redundancy have been completely observed by the authors.

LIFE SCIENCE REPORTING

No life science threat was practiced in this research.

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