



Comparison of the gaseous benzene adsorption capacity by activated carbons from *Fraxinus excelsior* L. as a lignocellulosic residual

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Abstract

Benzene vapor is notoriously known to induce adverse human health, which plays a definite role in the deformation of cells. Various advanced adsorbents from lignocellulosic precursors have emerged as cheaper alternatives to the green process for the adsorption of gaseous benzene. In this paper, the prominence mainly benzene vapor removal with lignin-based adsorbent in the adsorption technology has investigated the textural, morphology, and chemical characteristics of activated carbon synthesized from *Fraxinus excelsior* L. seeds, a lignocellulosic biomass waste. Chemically starting HCl and KOH in the N₂ atmosphere was adopted, contributing to the porous carbon material's well-developed porosity and surface chemistry. Also, carbonaceous materials were investigated by Brunauer–Emmet–Teller (BET), Fourier transform infrared (FTIR), scanning electron microscopy (SEM), and X-ray diffraction. Herein, the optimum way for producing activated carbon was recognized to be: activation temperature of 800 and 700 °C in line with an impregnation weight ratio of samples to HCl 1:2 and KOH 1:3 for 2 h activation time as FE₇AC and FE₂₂AC, which have resulted in 676 m²/g and 0.39 cm³/g; 734 m²/g and 0.48 cm³/g of BET surface area and total pore volume, respectively. The SEM observations exhibited advanced high porosity development formed by oxidation–reduction reaction, while FTIR confirmed the presence of various surface functional groups. Moreover, benzene became more tremendously facile for four ambient temperatures (20, 25, 30, and 35 °C) and until 200 min contact time. The tremendous values varied from 96 to 224 mg/g and 122 to 286 mg/g depending on lignin-based adsorbent amounts as 0.5 or 1 g and an initial benzene concentration of 100 mg/m³ by using FE₇AC and FE₂₂AC. The main innovation of this paper is exciting to assist recent paths for optimizing air filtration procedures under actual environmental circumstances, particularly regarding its compatibility with the benzene molecular structure of FE₂₂AC. The paper concludes that the performance of the FE₂₂AC can be enhanced via improvements in its surface properties for a wide array of actual benzene concentrations from gaseous applications.

Keywords Adsorption · Biosorbent · Chemical activation · Forest residues · Gas treatment

Introduction

The rapid decline in air quality was manifested by industrialization and urbanization problems (Isinkaralar and Varol 2023; Yao et al. 2023). Exposure to benzene is the forefront and abundant volatile organic compounds (VOCs) that emerged as organic solvents in the materials and chemical contents. The growth of human societies and the development of industries lead to monomeric hydrocarbons release

into the subsurface deliberately or accidentally, especially in developing countries (Isinkaralar et al. 2022; Pal et al. 2022). Continued growth in benzene emissions is a very worrying situation regarding environmental development and human health because it is onerous to decompose and can transport long distances (Liu et al. 2019; Zhao et al. 2022). Benzene, an odorous compound, can harm human health, manifesting in various dimensions with a concentration range of its environment (Chaiklieng et al. 2019; Nayek and Padhy 2020). A string of international environmental protection organizations' declarations has been widely reported to reveal benzene exposure type and concentration by Ministry for the Environment South Korea (2010), WHO (2010), ENVIRON (2014), and the Department of Environmental Health (2017) as it is a significant contributor to atmospheric

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pollution. Here, they explained benzene exposure results in all details and even described the adventure ranging from cancer to death at long-term exposure to the ubiquitous use of benzene-containing (Sakizadeh 2019; Spatari et al. 2021). Thus, mitigating or adequately controlling the gaseous benzene, a rapidly active, tremendous substance, is essential among the VOCs.

Benzene treatments have been studied in recovery techniques. However, they still need improving from the gaseous environment, lowering process costs and safety, and the conclusions drawn from separation or/and removal have necessary implications for the development of capturing benzene molecules with several materials. In fact, definitive mechanisms to remove have already been employed, such as membrane filtration (Kim et al. 2021a), photocatalytic degradation (Luo et al. 2022), biofilters (Rene et al. 2015), catalytic oxidation (Deng et al. 2018), non-thermal plasma (Saleem et al. 2019) and adsorption (Isinkaralar 2022a, 2023a), respectively. However, an important point is the presence of benzene gas, which may increase its intensity with many factors such as indoor-outdoor environment (Sekar et al. 2019), amount of presence (Liu et al. 2020), the form of presence (gas, liquid or liquid–gas particles) (Zavala et al. 2020), whether it is single or multiple (i.e., there are other volatile organic compounds at the same time) (Fetisov et al. 2023), ambient ventilation (Brdarić et al. 2019), ambient temperature–pressure (Jang et al. 2015), and the humidity of the environment (Li et al. 2020a). In this case, it is crucial to determine the removal method of benzene, which is desired to be eliminated, by evaluating the situation and considering the conditions. The adsorption method is known to be successful in VOCs environments (nonpolar) but not in all gaseous environments (polar) (Yang et al. 2018). However, the usage network for adsorption is quite rich, more simple, cost-effective, and eco-friendly among the methods (Miller et al. 2022). When the particular application results for liquid, gas, and liquid–gas in the literature were examined, the yield was relatively high compared to other methods (Zhang et al. 2017). Although the results showed the success of the adsorption process in a promising way, they also brought different results (Kwiatkowski and Broniek 2017; Szulejko et al. 2019). At the beginning of these, a wide range of conventional adsorbent materials used regular activated carbons (ACs) (Isinkaralar 2023b), MOF (Mehralipour et al. 2022), modified ACs (Deng et al. 2021), polymeric (Long et al. 2012), carbon nanostructures (Padilla et al. 2018) for separation or analytical purposes of benzene removal. The adsorbents mentioned above have advantages as well as disadvantages that, in turn, lead to the demand, such as the scarcity of raw materials used in the production phase, their value owing to minimum operational and maintaining cost, suitability of powder-granule-particle size, variability of pore volumes, number of desorption

cycles-efficiency, and the hazardous waste that arises in case of not being able to desorption (Gao et al. 2020; Quesada et al. 2020). The main point to know here is the correct selection of the application area and way with an optimum adsorbed amount. The high flow rate in industrial applications can cause higher adsorbent doses during the increased sharp fill-up to pores (González-García 2018; Pui et al. 2019). For this purpose, the high surface area and micropore volume of the produced ACs is a priority factor thanks to adequate installation or operation maintenance (Zhu et al. 2020; Zhang et al. 2022). In addition, it is vital to have suitable raw materials (abundant and cheap) and low activation costs to optimal produce in large quantities. In this case, the resulting agricultural, forestry, and farming wastes have proven themselves as appropriate raw material in the production of AC (Largitte et al. 2016; Ma et al. 2018; Franco et al. 2021). In particular, the authorities have accepted activating lignin-containing raw materials as environmentally friendly, green, and sustainable. *Fraxinus excelsior* L. are deciduous trees that have shown high production of valuable biomass for ACs production, which have been widely distributed in many geographies and have been treated in traditional medicine in different parts of the world (Flanagan et al. 2013; Isinkaralar 2023c).

In this study, it has been a priority for renewable carbon materials from seeds of *F. excelsior* for the lignocellulosic biomass and its incredible versatility of applications in benzene removal. It is aimed to obtain ACs using its seeds as a precursor. It needs the highest surface areas and pore volumes due to mixing and impregnating HCl and KOH, which are used in chemical activation at various ratios. To further clarify the physicochemical properties of ACs, this study was investigated, and the objective was to research the removal efficiency of benzene vapor at several concentrations. By comparing the yield, it has been tried to show how the effect of surface area and pore volumes could be significantly developed and utilized for benzene molecular retention. In addition, it is thought that the suitable chemicals treated into lignocellulosic material will lead to minimizing gas removal studies and inspire future VOC studies.

Materials and methods

Reagents and instruments

The lignocellulosic waste samples were collected as a forest residual during autumn from a local forest in Kastamonu, Türkiye. The proximal, ultimate, and lignocellulosic analysis seeds of *Fraxinus excelsior* L. were determined by ASTM standards. In the cumulative representation, it can be gained as its low content of ash (ASTM D 1102-84, 2013), moisture (ASTM E871-82, 2019), and high rate of volatile matter

(ASTM E872-82, 2019; ASTM 2011) indicates that the final carbonaceous *F. excelsior* seeds structure is corresponding to produce FEACs. Chemical activation of seeds was carried out with the use of hydrochloric acid (HCl) and potassium hydroxide (KOH) as activating agents, which were purchased from Merck (analytical grade). Sigma-Aldrich supplied benzene (99.8%) and BTEX mixture analytical standard, and Tenax®-TA single sorbent tubes (200 mg, 60/80 mesh Supelco, USA) were also provided from Millipore/Sigma. Nitrogen (N₂, 99.99%) and distilled water with conductivity < 1 μS/cm were also utilized in this experiment.

Post and in-situ synthesis of FEACs

The collected raw material was brought to the laboratory, and preliminary preparations for the synthesis processes were started to reduce to approximately 1 × 1 cm in size. Samples are purified abundantly and dried for one week at 45 °C to remove humidity. After making sure that it dries, it is separated into tiny particles, which are ground as the powder form. In order to eliminate the remaining moisture in the powdered samples, they were poured into a long tray, and the spread samples were mixed every hour and dried at 60 °C for 24 h. Each piece was weighed and prepared as 50 g into beakers. It was mixed with HCl at weight ratios of 1:1, 1:2, and 1:3, respectively. Meanwhile, it was heated at 100 °C for one hour with a continuous rotation of 150 rpm for 1 h. After being taken from the heater, it evaporated for a day at 105 °C. A horizontal tube reactor with a diameter of 8 cm and a height of 22 cm was utilized, accompanied by a nitrogen (N₂) flow rate of 80 mL/min as a carrier gas in a programmable furnace. At the beginning of the carbonization process, the temperature, N₂ flow, and digital indicators of the horizontal tube furnace were controlled in experiments. The solid form samples in the beaker were provided with uninterrupted pyrolysis in an N₂ environment at 600, 700, 800, and 900 °C with a heating rate of approximately 10 °C/min for 2 h. After 2 h, the furnace, left to cool without cutting the nitrogen supplied to the system, was opened after 4 h. The pH value of the activated samples obtained was neutralized by washing with 0.1 M NaOH. Then it was washed with boiling water to remove remnants and left in the oven at 105 °C to dry, then segregated using 0.45 μm of membrane filters. After performing the same procedures for KOH, it was washed with 0.1 M HCl to neutralize the pH value, then passed through the boiling water, filtered, and left in the oven at 105 °C to dry. Table 1 summarizes that a total of 24 FEACs are labeled from FE₁AC to FE₂₄AC and made ready for characterization.

The FE₇AC and FE₂₂AC were selected to investigation structural characteristics by BET (NOVA touch LX4, Quantachrome, USA) equation at the amount of N₂ adsorbed at the relative pressure $P/P_0=0.995$ for surface area calculation

Table 1 Nomenclature of FEACs

Impregnation ratio	Activating reagents	Ambient temperature	ACs ID
1:1 (w/w)	HCl	600 °C	FE ₁ AC
1:1 (w/w)	HCl	700 °C	FE ₂ AC
1:1 (w/w)	HCl	800 °C	FE ₃ AC
1:1 (w/w)	HCl	900 °C	FE ₄ AC
1:2 (w/w)	HCl	600 °C	FE ₅ AC
1:2 (w/w)	HCl	700 °C	FE ₆ AC
1:2 (w/w)	HCl	800 °C	FE ₇ AC
1:2 (w/w)	HCl	900 °C	FE ₈ AC
1:3 (w/w)	HCl	600 °C	FE ₉ AC
1:3 (w/w)	HCl	700 °C	FE ₁₀ AC
1:3 (w/w)	HCl	800 °C	FE ₁₁ AC
1:3 (w/w)	HCl	900 °C	FE ₁₂ AC
1:1 (w/w)	KOH	600 °C	FE ₁₃ AC
1:1 (w/w)	KOH	700 °C	FE ₁₄ AC
1:1 (w/w)	KOH	800 °C	FE ₁₅ AC
1:1 (w/w)	KOH	900 °C	FE ₁₆ AC
1:2 (w/w)	KOH	600 °C	FE ₁₇ AC
1:2 (w/w)	KOH	700 °C	FE ₁₈ AC
1:2 (w/w)	KOH	800 °C	FE ₁₉ AC
1:2 (w/w)	KOH	900 °C	FE ₂₀ AC
1:3 (w/w)	KOH	600 °C	FE ₂₁ AC
1:3 (w/w)	KOH	700 °C	FE ₂₂ AC
1:3 (w/w)	KOH	800 °C	FE ₂₃ AC
1:3 (w/w)	KOH	900 °C	FE ₂₄ AC

and DFT (density functional theory) applied to the adsorption isotherms for the pore distribution, SEM (Quanta FEG 250, FEI, USA) with 4500× magnitude and 10.00 kV for microstructures, FTIR (Perkin-Elmer TGA 7, Waltham, MA) using the KBr disk method (100 scans, from 4000 to 450 cm⁻¹, resolution 4 cm⁻¹) for surface chemistry, elemental analysis (EuroVector, model EA3000 Single, Italy) for elemental components, TGA (STA7300, Hitachi, Japan) under nitrogen and synthetic air atmospheres in an alumina crucible (from 25 to 1000 °C and heating rate of 20 °C min⁻¹) for decomposition processes and XRD (D8 Advance, Bruker AXS GmbH, Germany) with a CuKα radiation (40 kV, 30 mA and from 5 to 85) for determining the crystallite size and nitrogen adsorption (Quadrasorb-Tri, Quantachrome Instruments) at 77 K.

Benzene adsorption measurement

Before each test, FE₇AC and FE₂₂AC as carbonaceous adsorbents were separately premixed with benzene threshold concentrations from 5 to 100 mg/m³. 0.5 and 1 g of the FE₇AC and FE₂₂AC were used for each investigation. A batch reactor that allows the benzene to evaporate for sampling at

certain periods, temperature controlled, monitored the mass balance of benzene vapor is used for the experiment. It was connected to the N_2 flow rate of 50 mL/min as a carrier gas were directly collected into Tenax®-TA single sorbent tube by using a vacuum air pump (200 mL/min, AirLite 110–100, SKC, USA). Tenax tubes were analyzed using thermal desorption (TD; Markes Unity Series 2, Markes International Ltd., Llantrisant, U.K.) coupled with a gas chromatography/mass spectrometry detector (GC–MS; Thermo Scientific Trace 1300 and ISQ-QD Thermo Fisher Scientific). TG-624 capillary column (60 m × 0.25 mm ID × 1.4 μm film) for thermal desorber was inserted to check compounds based on US EPA Method TO-17 (USEPA 1999). To supply that the different test findings were calculated using Eq. (1) when FE₇AC and FE₂₂AC were occasioned by the amount of FE₇AC and FE₂₂AC rather than experimental errors, experiments under the same conditions were repeated at least once until reproducible results were attained.

$$q_{(mg/g)} = \left(\frac{FxC_0 \times 10^{-9}}{W} \right) \left[\left(\frac{C_i}{C_0} x t_s \right) - \left(\int_0^{t_s} \frac{C_i}{C_0} d_t \right) \right] \quad (1)$$

where q (mg/g), F (mL/min), W (g), C_0 (mg/m³), C_i (mg/m³), and t_s (min) are the saturated benzene adsorption capacity, the airspeed, the weight of FEACs, initial benzene concentration, benzene output concentration, and saturation time in removal system, respectively.

Results and discussion

The different physicochemical characteristics of FEB and FEACs were determined using ASTM standards.

Porosity properties of FEACs

Table 2 demonstrates the ultimate, proximal, and lignocellulosic analysis results of the selected materials, with the corresponding yield values. Cellulose hemicellulose and lignine values of 31.50 ± 3.40 , 35.80 ± 2.60 and 20.90 ± 3.80 in feedstock are the three main components of the biomass content of FEB. Also, near high volatile matter (68.53%), moisture (8.64%), and low ash content (1.12%) were found and they are dramatically lower than those of customarily used biomass materials. The carbon content of FE₇AC and FE₂₂AC were obtained as 67.27 and 78.22%. A somewhat higher yield was observed by increasing that carbon amount within a wide spectrum of applications tested. However, varying the impregnation ratio from 1 to 3 did not significantly affect that respect. Still, it increased the ash content of the resulting carbon due to more residual other elements remaining after washing. The elemental composition showed for FE₇AC and FE₂₂AC the carbon content as 67.27 and 78.22%, hydrogen as 4.96 and 1.34%, nitrogen as 2.83 and 0.77%, and oxygen as 21.42 and 19.21% thanks to the advantage of using lignin as the carbon precursor. It is a high grade precursor material for adsorption applications due to its high carbon content ($\geq 40\%$), cellulose (40% to 60%), hemicellulose (20% to 40%) and lignin (10% to 25%). (Boundzanga et al. 2022; Isinkaralar and Turkyilmaz 2022; Xiang et al. 2022). Arminda et al. (2021) determined the

Table 2 The result of the analysis of starting materials

Analysis type	Parameter	Selected materials		
		FEB	FE ₇ AC	FE ₂₂ AC
Ultimate	C (%)	44.07	67.27	78.22
	H (%)	4.58	4.96	1.34
	O (%)	49.36	21.42	19.21
	N (%)	0.39	2.83	0.77
	Others (%)	0.60	3.52	0.46
Proximal	Moisture (%)	8.64	2.05	2.59
	Volatile substance (%)	68.53	17.26	18.93
	Fixed carbon (%)	21.29	78.53	73.48
	Ash (%)	1.12	1.21	3.10
	Others (%)	0.42	3.00	4.49
Biochemical component	Cellulose (%)	31.50 ± 3.40	n.a	n.a
	Hemicellulose (%)	35.80 ± 2.60	n.a	n.a
	Lignin (%)	20.90 ± 3.80	n.a	n.a
Yield (%)		n.a	59.50	51.80

n.a. non-available

chemical composition of untreated olive tree pruning (OTP) residues. Accordingly, cellulose hemicellulose lignin was found to be 31.88%, 17.26% and 9.26%. However, these values changed to 17.73, 2.19 and 5.93 after pretreatment. Heidarinejad et al. (2020) reviewed porous carbonaceous material and unique adsorption properties that can be accelerated depending on structure diversity. The various lignocellulosic wastes have been converted into ACs for their variable values of lignocellulosic traits.

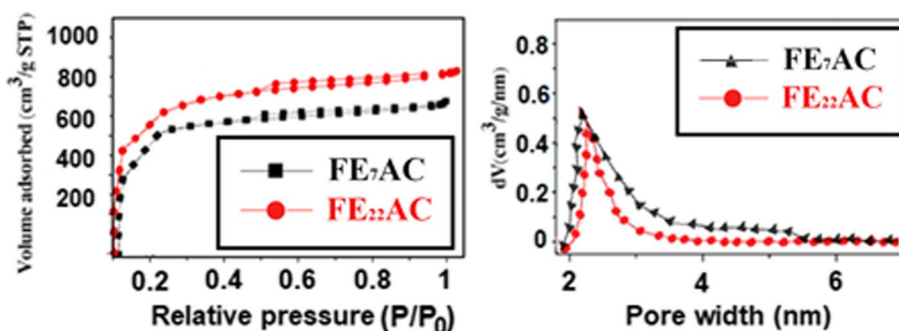
Figure 1 displays the N_2 adsorption/desorption isotherms and the pore distributions of the FE_7AC and $FE_{22}AC$. Figure 1A exhibits the isotherms hysteresis at a high relative pressure (P/P_0), mesopores characteristics, which the adsorption branch resembles that of a type I isotherm. Based on the International Union of Pure and Applied Chemistry (IUPAC) classification, Type I, the mostly microporous structure is crucial to highlight, indicating the materials have a strong affinity between adsorbent and adsorbate (Borhan et al. 2019). Figure 1B presents the mesopore size distribution for FE_7AC and $FE_{22}AC$, and the pore structure consists basically of micropores present 2–3 nm. These results are mainly defined predominantly in micropores together with a small external surface of FE_7AC and $FE_{22}AC$. Similarly, Yu et al. (2019) prepared TACs from tobacco stems by $ZnCl_2$ and TAC1, TAC3, TAC5, and TAC9 display a Type I isotherm as predominated by their micropores. El Nemr et al. (2022) studied to produce SBAC from sugarcane bagasse. They calculated its pore structure by N_2 adsorption isotherms correlated to Type I as microporous.

Surface morphology has an essential role in the activity of the FEB, FE_7AC , and $FE_{22}AC$ by using a scanning electron microscope. These results revealed the pores having the best performance and showed the developed natures of the FE_7AC and $FE_{22}AC$ after chemical activation compared to FEB. Figure 2A presents that FEB has no visible pores, although Fig. 2B and C shows irregular pore structures after HCl and KOH activation. Figure 2C shows higher porosity than Fig. 2B due to different preparation, indicating cavities and blanks. The surface area of the FE_7AC and $FE_{22}AC$ differed; however, there was no

difference in porosity because structural degradation with HCl has a higher effect than KOH disintegration. There is a comparable difference between FE_7AC and $FE_{22}AC$ during different synthesis forms. The precursors were impregnated by reacting chemicals, both acid and/or base, which occurs ACs with various morphological changes of final materials. Many SEM examinations revealed that ACs have a cavity surface with circularly shaped macropores of multiple sizes. If lignin-containing raw materials are used, ACs have rough characters, recessed and protruding porous (Contescu et al. 2018; Tsai and Jiang 2018; Mistar et al. 2020; Hassan et al. 2020). As reviewed in the literature for ACs obtained from lignocellulosic precursors, Abd-Rabboh et al. (2022) reported the well-developed pores, cavities, and heterogeneous amorphous surface texture on ACs from rice husk and straw wastes. According to Laksaci et al. (2017), ACs were synthesized carbonaceous materials using coffee grounds via KOH, namely ACK9, ACK18, and ACK36, which exhibit their heterogeneous surface and porous nature. Selvaraju and Bakar (2017) benefit from *Artocarpus integer* to develop AFP AC for adsorption applications. The character of AFP AC observed irregular pores-cracks and heterogeneous surfaces. From here, it is possible to know that the porous structure can be highly developed, although generally, they have non-homogeneous surfaces and pores.

Figure 3 displays the FTIR spectra of FEB, FE_7AC , and $FE_{22}AC$ which FEB comprises several functional groups such as the hydroxylic, carboxyl, and carbonyl groups. The broad absorption bands of FE_7AC and $FE_{22}AC$ for the presence of hydroxyl groups were observed at absorption peaks 3287 and 3183 cm^{-1} by stretching vibration of OH bonds (alcohols, phenols, and carboxylic acids). The presence of a wide peak between 2689 and 2704 cm^{-1} may be ascribed to the aliphatic CH stretching in alkyl groups. The bands between 1526 and 1587 cm^{-1} correspond to aromatic ring vibrations the for FE_7AC and $FE_{22}AC$, and the stretching band at 1072 and 1047 cm^{-1} was attributed to the vibration groups of (C–O) and (C–N) in hemicellulose and cellulose. It was also seen that FE_7AC and $FE_{22}AC$ obtained higher intensity adsorption bands compared to virgin

Fig. 1 N_2 adsorption–desorption isotherms at $-196\text{ }^\circ\text{C}$ of FE_7AC and $FE_{22}AC$ (A) and their pore size distribution (B)



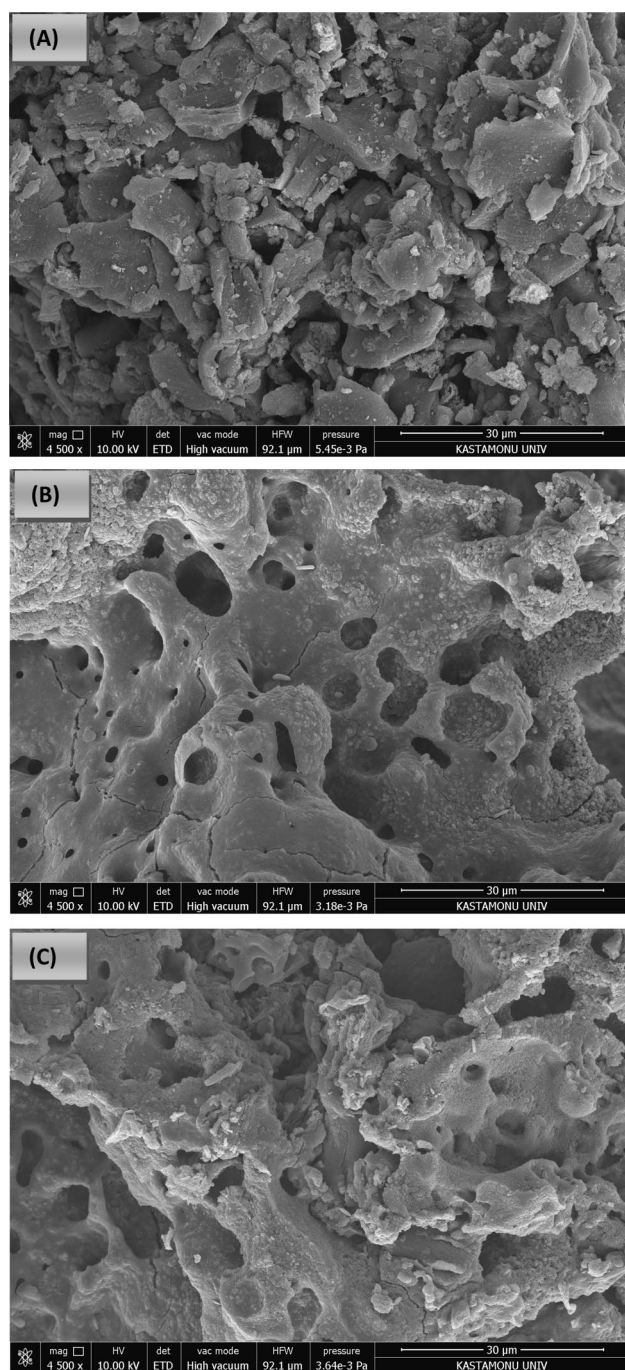


Fig. 2 SEM surface morphologies of FEB (A), FE₇AC (B), and FE₂₂AC (C)

FEB displaying increased amounts of carbon–oxygen and hydroxyl surface groups. The results of these studies are similar to those of other studies in which the formation of surface oxygen-containing functional groups on the adsorbent material which have played an essential role in the formation of some pores in the adsorption capacity by Zhao et al. (2020) and Tran et al. (2021).

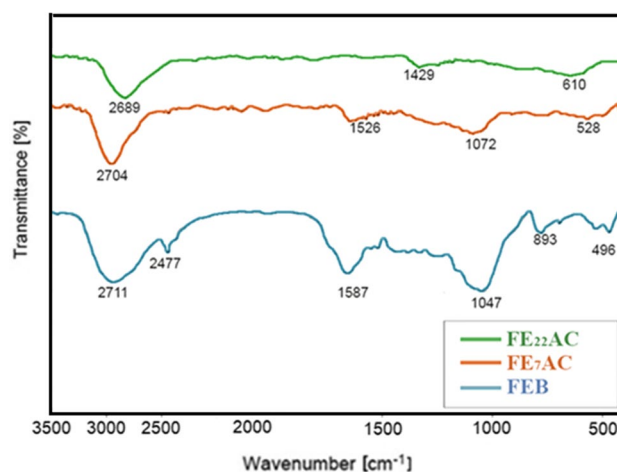


Fig. 3 FT-IR spectra of FEB, FE₇AC, and FE₂₂AC

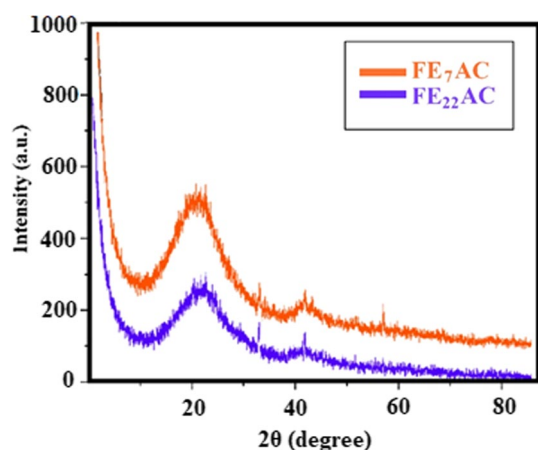
The FEACs were analyzed using Brunauer–Emmet–Teller (BET) analyzer to investigate the surface area, pore volume, and pore size, as shown in Table 3. Notably, the highest surface area, pore volume, and pore size of FE₇AC for HCl and FE₂₂AC for KOH were observed as 676 and 734 m²/g; 0.39 and 0.48 cm³/g and 2.53 and 1.97 nm, respectively. This is contributory to the HCl being impregnated on FEB; the pore size, surface area, and pore volume of the FE₇AC decreased compared to FE₂₂AC due to the difference in the action of HCl and KOH.

In several studies, the KOH activation method produced ACs with more microporosity than other chemicals (Wei et al. 2016; Li et al. 2017; Isinkaralar 2022b). For this purpose, Kharrazi et al. (2020) enhanced AC from an elm tree as a lignocellulosic waste sample for batch metal adsorption experiments. They tried to prepare chemical activation with KOH at 800 °C, inferred non-treated AC (465 m²/g), and thermal tension-treated AC (1085 m²/g). Samiyammal et al. (2022) used KOH to prepare AC for dye removal from cashew nut shells which obtained the highest surface area of 407.80 m²/g. Yang et al. (2022) emphasized that the KOH activation ratio of 1:1 (w/w) affects the preparation of AC from a by-product of low-rank coal by microwave method. The methylene blue adsorption was tried to remove using MKBC (their adsorbent name) effectively, and its surface area was reported as 857 m²/g. The significant difference of KOH activation is more effective than other activating agents, which results in more –OH functional groups on the surface of the ACs. In addition, potassium readily derives its carbon-intercalation compounds, thereby improving the adsorption mechanism of the ACs.

X-ray diffractogram of the fabricated FE₇AC and FE₂₂AC samples has been measured using X Powder of version 2004 graphic tool for powder diffraction in Fig. 4. The diffractograms pattern of FE₇AC and FE₂₂AC was impregnated by

Table 3 Surface areas and pore texture characteristics of FEACs

ACs ID	S_{BET} (m^2/g)	V_{total} (cm^3/g)	V_{micro} (cm^3/g)	V_{meso} (cm^3/g)	$V_{\text{micro}}/V_{\text{total}}$ (%)	$V_{\text{meso}}/V_{\text{total}}$ (%)	$D_p(\text{nm})$
FE ₁ AC	85	0.12	0.08	0.04	66.67	33.33	3.26
FE ₂ AC	109	0.18	0.11	0.07	61.11	38.89	3.80
FE ₃ AC	226	0.26	0.19	0.07	73.08	26.92	2.58
FE ₄ AC	334	0.29	0.21	0.08	72.41	27.59	2.67
FE ₅ AC	192	0.15	0.09	0.06	60.00	40.00	2.55
FE ₆ AC	351	0.24	0.14	0.10	58.33	41.67	2.90
FE ₇ AC	676	0.39	0.25	0.14	64.10	35.90	2.53
FE ₈ AC	588	0.33	0.23	0.10	69.70	30.30	3.51
FE ₉ AC	208	0.17	0.11	0.06	64.71	35.29	3.05
FE ₁₀ AC	293	0.29	0.23	0.06	79.31	20.69	2.73
FE ₁₁ AC	547	0.37	0.27	0.10	72.97	27.03	2.59
FE ₁₂ AC	521	0.36	0.25	0.11	69.44	30.56	2.44
FE ₁₃ AC	116	0.18	0.13	0.05	72.22	27.78	3.08
FE ₁₄ AC	238	0.25	0.16	0.09	64.00	36.00	3.42
FE ₁₅ AC	366	0.36	0.26	0.10	72.22	27.78	1.95
FE ₁₆ AC	458	0.33	0.20	0.13	60.61	39.39	2.43
FE ₁₇ AC	139	0.22	0.15	0.07	68.18	31.82	2.25
FE ₁₈ AC	308	0.29	0.22	0.07	75.86	24.14	2.61
FE ₁₉ AC	445	0.38	0.29	0.09	76.32	23.68	2.40
FE ₂₀ AC	543	0.33	0.25	0.08	75.76	24.24	3.48
FE ₂₁ AC	267	0.26	0.18	0.08	69.23	30.77	2.75
FE ₂₂ AC	734	0.48	0.34	0.14	70.83	29.17	1.97
FE ₂₃ AC	630	0.39	0.33	0.06	84.62	15.38	1.35
FE ₂₄ AC	557	0.44	0.32	0.12	72.73	27.27	2.17

**Fig. 4** XRD patterns of FE₇AC and FE₂₂AC

HCl and KOH at different activation temperatures and times. The apparent peaks: the first cluster provided 2θ X-ray diffractogram from 5° to 10° , the second one provided peaks at 22.3° and 24.5° , and the third one attained peaks of FE₇AC is detected 34.5° and 57.2° . XRD analysis of FE₂₂AC depicts 32.6° and 41.8° . The above results tell you how high the relative quantity and density of atomic clusters is. It also clearly

shows that there are more atoms. The activating of FEB leads to the substantial degradation of lignocellulosic material. The latter accelerates peak intensities of the FE₇AC, and FE₂₂AC has a higher carbon yield than FEB due to higher activation temperature and longer activation time. Mohammed et al. (2020) studied XRD analysis of TPAC from tangerine peel and reported similar results. The broad diffraction peaks between 25.18° and 45.85° confirm the presence of graphite crystallite in TPAC is the expected structure for its adsorption mechanism. Chouikhi et al. (2021) peaks were observed in biochars and ACs, which were attained at diffraction angles (2θ) with corresponding planes between 25° and 44° . In many cases, some researchers show similar successfully formed and supported chemical bonds between their biomass during carbonization on the crystallinity of the ACs by Tiegam et al. (2021) and Xue et al. (2022).

The thermal decomposition of FEB, FE₇AC, and FE₂₂AC under an N₂ atmosphere is shown in Fig. 5. In the samples of the three materials, the TGA weight loss curves define three main stages typical for FEB pyrolysis. The first stage corresponds to moisture release, roughly examined at $35\text{--}190^\circ\text{C}$. The remaining two steps are connected to active and passive pyrolysis; the most significant mass loss characterizes the active stage. Herein, various vapors and gases are released

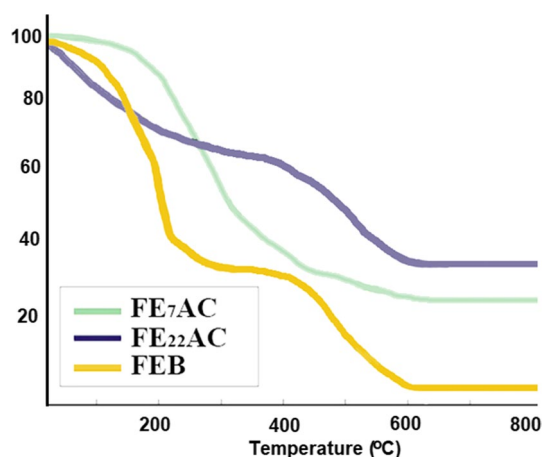


Fig. 5 Thermal stability analysis of FEB, FE_7AC , and $FE_{22}AC$

due to the decomposition processes of hemicelluloses and cellulose, which happen in this stage between 190–440 °C and 450–580 °C, respectively. Shrestha and Rajbhandari (2021) showed weight loss of their raw material, which exhibited significant weight loss at 300–400 °C due to the breakdown of cellulose into components. In addition, the TGA curve revealed that all FE_7AC and $FE_{22}AC$ were thermally stable up to 850 °C. It can be observed that with an

increase in the heating rate that the mass losses occur at increasing temperatures. This behavior has been described by (Alghamdi et al. 2019) and (Hossain et al. 2022) can be explained using different arguments. The thermo-gravimetric curves highlighting the mentioned losses of mass can be achieved, suggesting that chemical activation with HCl and KOH resulted in a considerable degree of functional formation on the excellent thermal stability of FE_7AC and $FE_{22}AC$.

Adsorption of benzene vapor

Saturated adsorption capacity

Adsorption studies were carried out at operating temperatures of 20, 25, 30, and 35 °C. The amount of adsorbent used was loaded into the system as 0.5 and 1 g. System operation was started without adsorbent and was monitored until it became stable. Then the different concentration amounts were given to the system; From 5 to 100, the effects of adsorbents were investigated, and although the amount doubled, the adsorbed amount did not double. Based on Fig. 6, the values obtained for FE_7AC and $FE_{22}AC$ at 0.5 and 1 g, up to 100 mg/m^3 ; (i) 132 and 193 mg/g at 20 °C operating conditions; 151 and 237 mg/g , (ii) 146 and 224 mg/g at

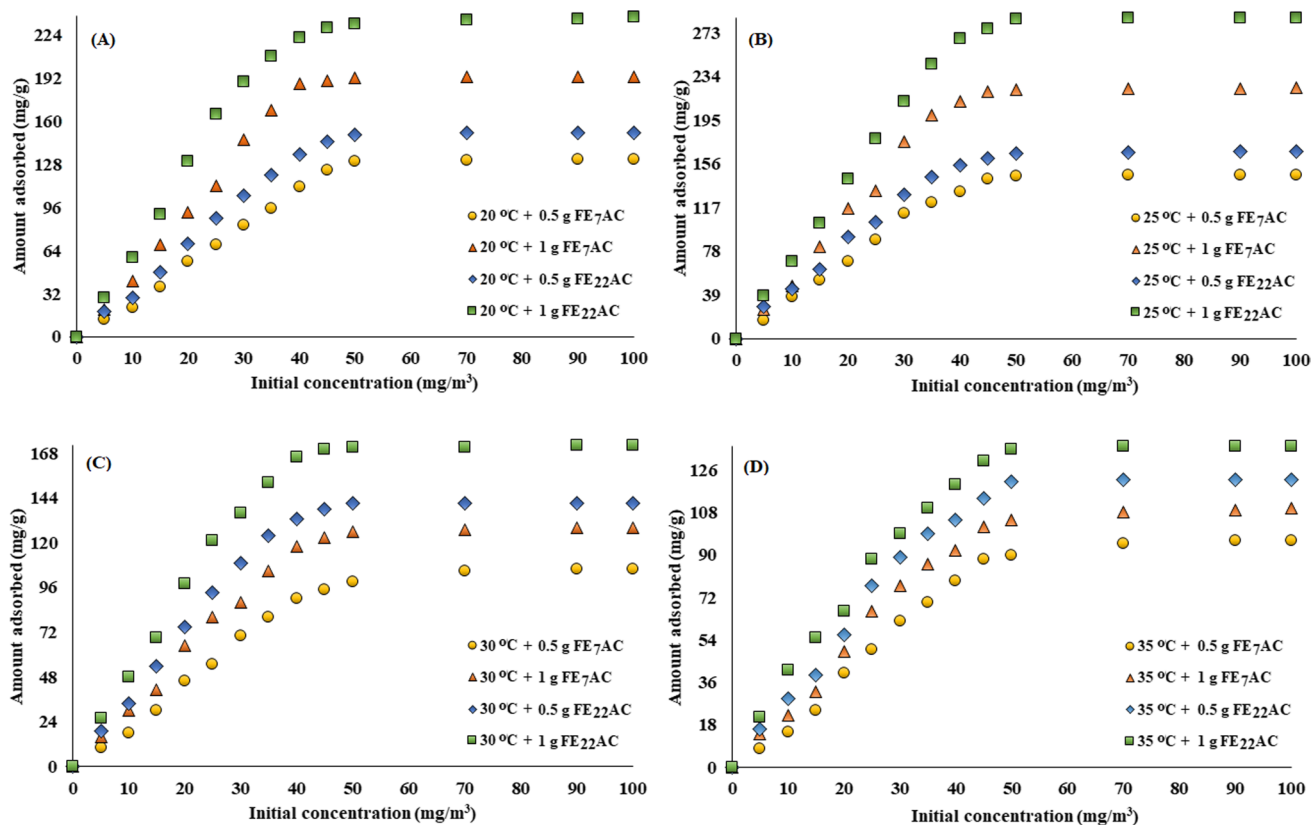


Fig. 6 Steam temperatures (A): 20 °C, (B): 25 °C, (C): 30 °C and (D): 35 °C for different initial concentrations on the different amounts of FE_7AC and $FE_{22}AC$

25 °C operating conditions; 167 and 286 mg/g, (iii) 106 and 128 mg/g at 30 °C operating conditions; 141 and 172 mg/g, (iv) 96 and 110 mg/g at 35 °C operating conditions; 122 and 136 mg/g. The increase in temperature caused an increase in the adsorption capacity up to 25 °C and then a decrease in the amount of adsorption when it reached 35 °C. The authors indicated that the experiments emphasized positive correlations between benzene molecules and increased initial benzene concentration (Moussavi et al. 2013; Konggidinata et al. 2017; Valencia et al. 2022).

Experimental studies report that the adsorption ability with carbonaceous samples has been affected not only by their prepared methods but also has been impressed, such as existing benzene concentrations by Mataji and Khoshandam 2014; Mohammed et al. 2015 and de Mello et al. 2022. Relatedly, Shi et al. (2022) investigated the gaseous benzene adsorption onto AC7 (S_{BET} : 2669.40 m²/g) under initial concentrations (1000–5000 ppm) and environmental temperatures (25, 35, and 45 °C). They proved that the driving force of mass transfer was raised due to the growth of the initial concentration. Stähelin et al. (2018) measured the maximum adsorption capacity of benzene in both monocomponent and monocomponent processes. The bicomponent and monocomponent systems were operated with several initial

benzenes from 56 to 450 mmol/L and 113 to 450 mmol/L onto ACC from coconut shell. Benzene concentration at higher initial values increases the adsorption capacity. He et al. (2021) utilized PAC-5–850-60 to remove gaseous benzene, which was carried out to explore the maximum diffusion of benzene molecules with the increase in the initial concentration (from 100 to 5000 ppm). The maximum adsorption capacity for 100, 1000, and 5000 ppm was calculated at 122.19, 437.11, and 819.77 mg/g for PAC-5–850-60.

Adsorption equilibrium time

The adsorbent amount was fixed at 0.5 and 1 g, and the adsorption capacities up to 200 min were compared by increasing the system's temperature. Plausibly, the results were compared for 0.5 and 1 g FE₇AC and FE₂₂AC was injected with 100 mg/m³ benzene as an initial concentration. Respectively, when the values obtained for FE₇AC and FE₂₂AC at 20, 25, 30, and 35 °C were compared, as shown in Fig. 7, they adsorbed more than 65% in the first 20 min. It was observed that the limit values were reached in adsorption capacities between 20 and 50 min. However, the adsorbents were expected to reach equilibrium conditions, and adsorption stopped at 200 min. This evidence suggests

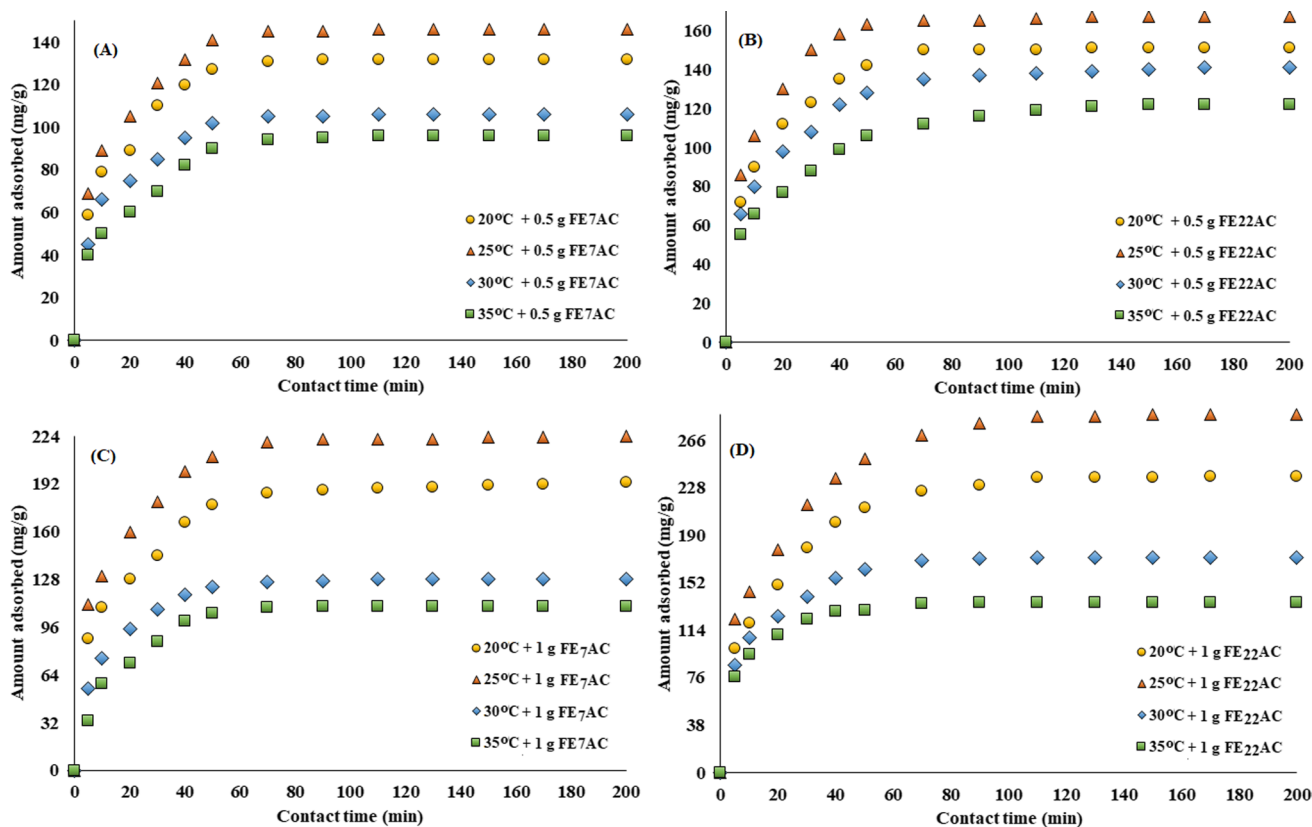


Fig. 7 Comparison of residence time curves on the different amounts (A): 0.5 g FE₇AC, (B): 0.5 g FE₂₂AC, (C): 1 g of FE₇AC and (D): 1 g of FE₂₂AC

a link between dramatically decreasing benzene adsorption amount and rising temperature effects on a potential loss of adsorptive benzene molecules due to their behaviors becoming more active at ≥ 25 °C. It is indispensable to be aware of the increasing temperature for benzene adsorption; thus, research results have been consistent with our findings. In Li et al. (2020a, b) research, the benzene adsorption rate investigated the filling pore process on carbon adsorbents (AC-1, AC-2, AC-3, and AC-4) under different temperatures (0, 15, 30, and 45 °C). Their results indicate that the adsorption amount of the AC-1 and AC-2 changed little with temperature; however, the adsorption values of the AC-3 decreased until 30 °C. The AC-3 obtained more excellent adsorption due to its larger micropores than others. Chen et al. (2023) systematically employed dynamic benzene adsorption on hollow carbon spheres (HCS-11 S_{BET} : 2531.29 m²/g and S_{Micro} : 3.527 m²/g) and commercial coal-based AC (CBAC S_{BET} : 1109.04 m²/g and S_{Micro} : 0.459 m²/g) at 20, 30, and 40 °C. The saturated adsorption capacities compared between HCS-11 and CBAC at 20, 30, and 40 °C were 21.363–5.889, 20.389–5.716, and 19.981–5.617 mmol/g. The results appeared the adsorption capacity slowed down with increased temperature. Also, the CBAC showed a relatively minor adsorption amount than HCS-11 due to a smaller proportion of micropores. Several researchers indicate positive correlations between benzene rings and the increase in temperature (Saleh and Danmaliki 2016; Deng et al. 2021; Jareteg et al. 2021).

Adsorption technology has been widely used to prevent the problems caused by exposure to benzene gas at different concentrations. In addition, some literature has reported that the production and modification of various adsorbents increased, accelerating the adsorption efficiency (Martínez de Yuso et al. 2013; Dizbay-Onat et al. 2018; Gayathiri et al. 2022). While the increases were realized, the production phase was left behind regarding environmentalist, sustainability, and cost–benefit. However, the production steps of the lignin-containing adsorbent used in this study are quite reasonable, low-budget, easy to apply, high in surface area and pore volume, and include environmentally friendly approaches. Moreover, a large number of initiatives have produced adsorbents with cheap, renewable (Saadi et al. 2022) and abundant biomass (İlbay et al. 2017), but they have removed pollutants in liquid media, not in gaseous media (Karimnezhad et al. 2014; Anand et al. 2021). Although the research is limited to the gaseous environment, some people have made different VOC removals (Khan et al. 2019; Ma et al. 2021). The effective point in these studies is the simultaneous multiple degassing or a single type of degassing. An example of these efforts is the effectiveness of adsorbents used in multiple degassing is much less than those used in single degassing (Osuchowski et al. 2019). In systems working as multi-component, different molecular

structures entering the pores of the adsorbent and their filling of the pores are in a dispersed, unsystematic form without being homogeneous; in a system, which works as a single component, it is noteworthy that the molecules filling the pores are homogeneous, more systematic and stable (Lee et al. 2019). In particular, the filling of the pores of several adsorbents occurs differently from each other (Feng et al. 2023). The homogeneous structure of the pores is seen to be less in number, although it is more systematic while taking in the molecules. However, studies have shown that the heterogeneous structure obtained after activating lignin-containing structures includes benzene molecules (Saha et al. 2018). Based on these studies, general judgments can be reached by comparison measurements under different conditions, as differences arise in terms of characteristics such as reactor type (discrete, continuous, etc.) (Azhagapillai et al. 2021), adsorbent structure and amount (Kim et al. 2021b), multi- or single component (Ouzzine et al. 2019), the molecular structure of the gas to be removed, ambient temperature-humidity (Oh et al. 2019), and contact time (Qiao et al. 2021).

Conclusion

Developing the surface area on the gaseous benzene removal efficiency is a vital recoverer to indoor air pollution. Indoor air quality must be unquestionably good in areas that cannot be ventilated, in public transportation, indoors, shopping malls, vehicle and air conditioner filters, health centers, and schools. The production of FE₂₂AC with high adsorption capacity using an economical and straightforward technique can still be a great subject to inspect biomass transformation into carbonaceous materials by chemical activation. The adsorbing ability of the produced activated carbon up to 200 min was examined, and the results were exhibited on its adsorptive potential. A microporous FE₂₂AC having a high surface area and total pore volume (676 m²/g and 0.39 cm³/g) resulted from KOH impregnation under 1:3 (w/w) carbonized at 700 °C for 2 h of activation time. The deposited directly onto the FE₂₂AC from the gaseous environment resulted in its highest potential of 286 mg/g at the ambient temperature of 25 °C at an initial benzene concentration of 100 mg/m³ and for 200 min. Another powerful adsorbent is FE₇AC which has a lower adsorption capacity than FE₂₂AC because of its physical and chemical characteristics. This would specifically help to reveal economic and eco-friendly gas benzene capture mechanism was found desirable for good biosorption potential and reuse of recycled materials.

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Availability of data and materials The data that support the findings of this study are available from the corresponding author, upon reasonable request.

Declarations

Conflict of interest The author declares no competing interests.

Ethical approval Not applicable.

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