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ORIGINAL ARTICLE

Does agricultural biomass matter for environmental sustainability? Enhanced adsorption capacity of BTEX mixture using powdered activated carbon by agricultural biomass

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Abstract

The poor indoor air quality can be associated with the released volatile organic compounds (VOCs) from different sources. The extent of the concern may increase depending on the presence of benzene, toluene, ethylbenzene, and xylene (BTEX) and exposure to them in the indoor air. Adsorption with activated carbon, which is a very effective method, is preferred to eliminate highly volatile gaseous pollutants and reduce the extend of their negative impact. In this work, the removal efficiency of a novel activated carbons (MSRACs), prepared from stems of *Corylus colurna* (CCBW) by chemical processes using H_2SO_4, H_3PO_4, and HCl, was scrutinized towards BTEX pollutants. The adsorbents acquired from this lignin-based waste were investigated from porosity and surface chemistry aspects. The highest surface area of 1424 m^2/g and micropore volume of 0.46 cm^3/g were attained after activation of MSRAC11 adsorbent sample by H_2SO_4-70wt%. The performances of the fabricated adsorbent samples were evaluated and the order of MSRAC11>MSRAC24>MSRAC36 was obtained in the multiple concentrations of BTEX. This study introduces an easy method for producing efficient adsorbents from lignin-based waste for filtering indoor air and designing BTEX-capturing systems for various applications.

Keywords Sorption · Agricultural waste · Gas-phase · Lignin-based · Sorbent production

1 Introduction

Benzene, toluene, ethylbenzene, and xylene (BTEX) are mainly anthropogenic pollutants which are emitted from various areas [1–3]. BTEX is released from several sources, such as gas stations, solvent production, smoking, cleaning agents, polish adhesives, dyestuffs, and wood production industry [4–6]. Moreover, they are frequently found in the indoor microenvironment, including building materials [7, 8]. BTEX is classified as dangerous and toxic in terms of quality-quantity and adversely affects public health at high exposure levels, by destroying human DNA [9–11]. Benzene is one of the most investigated prioritized pollutants which have categorized as human carcinogens by the International Agency for Research on Cancer [12]. Toluene and ethylbenzene can show toxicity, are capable to bioaccumulate in the human body, and cause damage to the central nervous system, and xylene causes skin and respiratory tract damage [13, 14]. So, monoaromatic hydrocarbons' expected effect is neurotoxicity in the cases of their presence in the environment [15, 16].

The reactions of BTEX in the atmospheric chemistry lead to production of some photochemical precursors [17]. Tropospheric ozone and secondary organic aerosol gases can be produced due to highly reactivity of BTEX pollutants in the troposphere [18]. Several institutions, countries, and environmental organizations, such as the World Health Organization (WHO) and the US Environmental Protection Agency (EPA), make various measurements (in outdoor and indoor environments) to control BTEX's presence and estimate its effect on the indoor environment worldwide. Especially in indoor areas with high emission potential (such as industry and chemical building materials), the measurements are necessary to continue as spatial and

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temporal variations of BTEX compounds [19, 20]. The main reason for the high BTEX ratio of the air in polluted area is that it rapidly evaporates at room temperature [21, 22]. They are released into the atmosphere, which allows interpretation of the indicator of the atmospheric photochemical activity by toluene/benzene (T/B) and ethylbenzene/m,p-xylene (E/X) [23, 24]. Considering the areas of use of BTEX gases, the alarming picture emerges as the indoor environment's duration and amount of exposure. Increased fatigue, headaches, weakness, loss of appetite, focusing problems, and many other health symptoms are observed in humans [25, 26]. Additionally, the BTEX amount in some indoor environments is relatively high compared to other contaminants, which is caused by using coal [27, 28], insufficient or inadequate ventilation [29, 30], operating photocopy and fax machines [31], paper and cardboard recycling [32], automobile-related reactions [33, 34], hospitals [35], sick building syndrome (SBS) in buildings [36, 37], and petroleum stations [38]. Hence, people want a decrease in BTEX exposure, especially in the more concentrated residence areas, because they feel anxiety about the high amount of such contaminants in some living areas.

In addition, recent studies have focused on some methods and their applications to remove BTEXs from the environment [39–42]. The adsorption method has achieved high success and efficiency using powdered or granular activated carbons [43–45]. Fortunately, the material with any carbon content can be prepared via suitable preparation conditions. However, some raw materials (e.g., wood, nutshells and fruit stones, peat, charcoal, soft coal, lignite, bituminous coal, and petroleum coke) have high carbon content than lignocellulosic materials (such as agricultural wastes and woody plants) [46, 47]. The porous structure of activated carbons can also be improved by impregnation (such as preparation and evaluation of fine-tuned micropore biochar by lignin impregnation for CO_2 and VOC adsorption) and ball milling (such as effect of ball milling with hydrogen peroxide or ammonia hydroxide on sorption performance of volatile organic compounds by biochar) [48, 49]. While the dehydration of the raw material at different temperatures is aimed with pyrolysis, at this stage, the aromatic content of the carbon skeleton and charring cause the pores to be formed [50, 51]. In the prepared sorbents, O, H, and C content of the prepared sorbents bears some information about functional surface groups on the adsorption properties [52]. Although BTEX monitoring studies are relatively high, BTEX removal has been limited and is a very current topic. It has been revealed that BTEX capture studies are troublesome because they evaporate and show quick behavior at room temperature. Many studies have been carried out on the behavior of BTEX molecules at various temperatures considering the binder types [53, 54].

In this research, activated carbons (MSRACs) are produced from the stems of *Corylus colurna* (CCBW) by chemical activation with H_2SO_4, H_3PO_4, and HCl. The stem of *Corylus colurna* was selected since it is an abundant lignocellulosic waste. The produced MSRACs were employed for BTEX capture in a controlled closed system, and the influential variables were assessed and optimized. In fact, *Corylus colurna* stem wastes, by using different activation agents and optimizing the process conditions, were exploited for the possibility of effective reduction of levels of gas-phase BTEX which was comprehensively investigated and reported.

2 Material and methods

2.1 Materials and reagents

Corylus colurna stem was obtained from a garden in Kastamonu, Turkey. The collected biomass was washed with plenty of water and left to dry. After being reduced to specific dimensions (it has a diameter of 3–4-cm long), it was ground with the help of a grinder and sieved. The activation agents were H_2SO_4, H_3PO_4, and HCl solutions. The reagents were analytical grade and used as received. Also, the solutions were prepared with deionized water throughout the experiments.

2.2 Precursor and MSRAC preparation

For the carbonization process of the ground *Corylus colurna* stem, (i) nitrogen gas (N_2) with a flow rate of 50 cm^3/min was given to the environment in the high-temperature resistant carbonization reactor, 550 °C, 5 °C per minute, and left to cool after standing in the reactor for 1 h; (ii) 30 g of the ground stem of *Corylus colurna* powder was mixed with a ratio of 10 wt%, 30 wt%, and 70 wt% with H_2SO_4 (agent 1), H_3PO_4 (agent 2), and HCl (agent 3) chemicals; (iii) after mixing the mixture until homogeneousness, increasing 5 °C per minute, the activation process was accomplished, exposing the precursor to the temperatures of 550 °C, 650 °C, 750 °C, and 850 °C for 80 min in the presence of N_2 gas; (iv) After 80 min, N_2 gas was continued to be injected into the system until the reactor temperature dropped to room temperatures; (v) pH level of the samples taken from the reactor was declined by mixing each sample with 0.1 M NaOH solution until it became neutral and washed with hot distilled water and kept at 70 °C for drying in the oven; (vi) the production efficiency was calculated by weighing the product; (vii) the activated carbons obtained are numbered in Table 1, and the removal processes were systematically started.

Table 1 Making a comparison among various MSRACs for better performance under several conditions

Added chemicals ratio	Pyrolysis stage temperature (°C)	Agent 1	Agent 2	Agent 3
10 wt%	550	MSRAC1	MSRAC13	MSRAC25
	650	MSRAC2	MSRAC14	MSRAC26
	750	MSRAC3	MSRAC15	MSRAC27
	850	MSRAC4	MSRAC16	MSRAC28
30 wt%	550	MSRAC5	MSRAC17	MSRAC29
	650	MSRAC6	MSRAC18	MSRAC30
	750	MSRAC7	MSRAC19	MSRAC31
	850	MSRAC8	MSRAC20	MSRAC32
70 wt%	550	MSRAC9	MSRAC21	MSRAC33
	650	MSRAC10	MSRAC22	MSRAC34
	750	MSRAC11	MSRAC23	MSRAC35
	850	MSRAC12	MSRAC24	MSRAC36

2.3 Characterization

The morphological structure of MSRACs was observed using scanning electron microscopy (SEM), and FTIR were used to search the surface functional groups on MSRACs. The specific surface area of MSRACs was calculated by the Brunauer-Emmett-Teller (BET) equation which was also used to obtain the pores volume of MSRACs [55].

2.4 Batch adsorption experiments

The sequential batch adsorption experiments for BTEX removal carried out at different sorption times, initial sorbate concentrations, humidity levels, and temperatures, according to indoor air conditions. Before starting the sorption, quality control experiments were checked recursively to maintain the reactor's stability of the BTEX concentration. The sequential batch reactor is made of glass quartz and has a thermostatic temperature and humidity meter to adjust the desired humidity and temperature. Additionally, removal efficiency studies were performed in triplicates by relative standard deviation or mean squared error in all experiments.

The BTEX mixture obtained by mixing in a single glass beaker was evaporated at 25 °C, without allowing any gas to escape into the indoor environment. The gas phase concentration in the steady-state conditions was set to be 30 $\mu g/m^3$, 70 $\mu g/m^3$, and 120 $\mu g/m^3$, respectively. After the sequence batch reactor was prepared in the system, the yield measurement results of 0.05 g MSRAC11, MSRAC24, and MSRAC36 were taken in 5-min periods starting from 0 to 45 min. The processes were tried at least three times to prevent contamination or leakage of the different starting amounts determined in the system. In the removal efficiency studies, the size of the pores and the pressure-humidity-temperature relationship should be known clearly. Experiments were carried out by taking the ambient conditions in the study, room temperature, and average humidity. Therefore, the variability of the mentioned factors was not tested.

Table 2 Elemental compositions of CCBW and MSRACs

Elemental analysis (%)	CCBW	MSRACs		
		MSRAC11	MSRAC24	MSRAC36
C	47.07	73.81	70.02	66.45
H	5.58	2.61	3.02	3.49
O	17.22	20.91	18.53	19.61
N	0.65	0.11	0.39	1.78
Other elements	29.48	2.56	8.04	8.67

3 Results

3.1 Physicochemical properties of MSRACs

The elemental composition analysis in the different production stages has been supplied to form the physicochemical properties of the adsorbent, including the impregnation ratio and the carbonization temperature. Measurements of each as different amounts of C, H, O, N, and other elements were traversed. The amount of C in the basic structure of the raw material increased from 47.07 to 73.81% in MSRAC11, the H ratio in the CCBW decreased from 5.58 to 2.61%, and the total amount of other elements in the structure before the carbonization process increased from 2.56 to 29.48% as listed in Table 2.

The impregnation ratios were compared to each other in Table 3. The highest yield and performance were obtained after mixing at 70wt%. Thus, since the BTEX sorption efficiency of 10wt% and 30wt% is predicted to be low, removal studies have started with 70wt%. The porosity (micro, meso, and macro) in the structure of MSRACs has been analyzed as one of their physicochemical parameters. The MSRAC11, MSRAC24, and MSRAC36 have the highest surface area, emerging among each agent, respectively, and these values were found as 1424 m^2/g, 1330 m^2/g, and 1342 m^2/g. The highest micropore volume of 0.46 cm^3/g was belonged to MSRAC11. Also, a surface area of 1409 m^2/g was obtained at 850 °C in MSRAC12, and its micropore volume was found to be 0.30±0.11 cm^3/g.

SEM images show that whether the pores in the structure before and after the study have changed. Figure 1A shows that the raw material has almost no pores, but after the chemical activation process, pores are included in Fig. 1B, and the structure is completely changed. The pores and micropores become more frequent, and the number of pores increases, as shown in Fig. 1C and Fig. 1D. It is noteworthy that the

Table 3 Mean, standard deviation, minimum, and maximum of BET surface area and pore volumes of MSRACs

Agents	Carbonized (°C)	Eco-friendly sorbent ID	Yield (carbonized basis) (%)	BET (m^2/g)			Micropore volume (cm^3/g)		
				Mean±SD	Min	Max	Mean±SD	Min	Max
1	550	MSRAC9	78.5	263±99	164	362	0.08±0.05	0.03	0.13
	650	MSRAC10	77.3	549±147	402	696	0.16±0.03	0.13	0.19
	750	MSRAC11	73.9	1024±608	416	1424	0.27±0.05	0.12	0.46
	850	MSRAC12	68.4	939±470	469	1409	0.30±0.11	0.19	0.41
2	550	MSRAC21	80.6	238±112	126	350	0.12±0.07	0.05	0.19
	650	MSRAC22	77.8	586±183	403	769	0.17±0.12	0.05	0.29
	750	MSRAC23	75.7	902±404	498	1306	0.23±0.10	0.13	0.33
	850	MSRAC24	70.1	967±313	654	1330	0.27±0.10	0.17	0.37
3	550	MSRAC33	81.3	151±105	46	256	0.15±0.08	0.07	0.23
	650	MSRAC34	76.5	367±106	261	473	0.23±0.12	0.11	0.35
	750	MSRAC35	73.6	681±155	526	836	0.29±0.16	0.13	0.45
	850	MSRAC36	71.2	947±395	552	1342	0.37±0.05	0.22	0.42

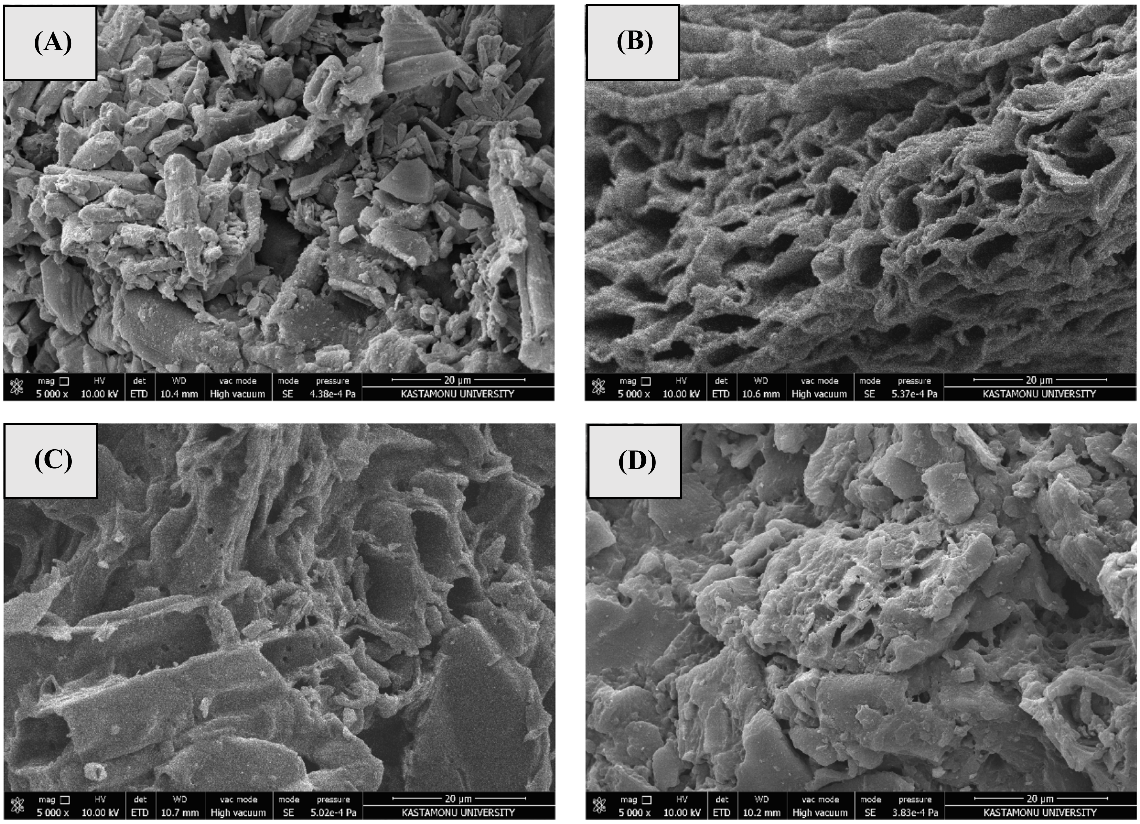

Fig. 1 SEM micrographs of **A** CCBW × 5000, **B** MSRAC11 × 5000, **C** MSRAC24 × 5000, and **D** MSRAC36 × 5000

inhomogeneous appearance in the structures is usual in activated carbons obtained from lignin-based materials. It has been stated in other studies that the surface distribution of lignin structures is not homogeneous.

Figure 2 shows FT-IR spectra and exhibits changes in some functional groups, such as carboxyl, carboxylic anhydride, phenol, and lactone on MSRACs. The surface stretching vibrations depend on the carbon matrix and the existing bonds between dissimilar of similar atoms such as H, C, N, and O [56]. There is a prominent peak at about 1421 cm^{-1} which can be assigned to vibrations of C=O and aromatic ring [57, 58]. Another region where the other peaks are concentrated is between 2900 and 3100 cm^{-1} which be dedicated to the in-plane bending vibration of

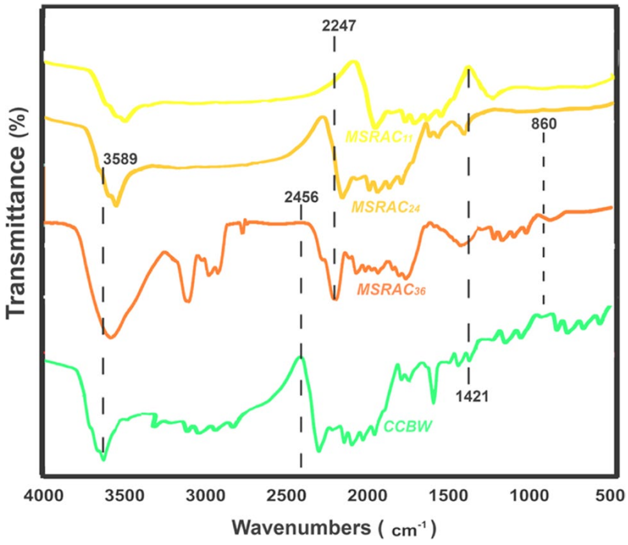

Fig. 2 FT-IR spectroscopy of CCBW, MSRAC11, MSRAC24, and MSRAC36

–OH or carboxyl carbonates structures and the stretching vibration N-H group, respectively. Values less than 800 cm^{-1} correspond to the fingerprints of the activated carbon structures [59]. Besides the significant peaks, small peaks at 3589 cm^{-1} represent the stretching vibration of the O-H bond of hydroxyl groups [60]. Also, we can see its presence in the range of 500–860 cm^{-1}.

3.2 Effect of experimental variables

Figure 3 shows the graph of the increasing concentration with the retention rates and the variability of their stable point. In Fig. 3A, the benzene removal efficiency was less than toluene but higher than ethylbenzene at 37.9 µg/g in MSRAC11. This sorbent has a higher adsorption capacity than the others, followed by MSRAC24, then MSRAC36. Among the volatile organic substances evaluated separately, xylene had the highest sorbed amount of 38.2 µg/g, while the lowest amount was belonged to ethylbenzene. While the sorption percentage was high in the first 20 min (Fig. 4), a decreasing curve was observed in this parameter afterward. The main reason can be the filling of the pores and the decrease in their holding capacity. At this point, the MSRACs become entirely saturated, and no adsorption occurs in the test. Consequently, once saturation time is reached, the outlet concentration is constant and equal to the residuary concentration.

The adsorption efficiency was compared to the initial concentration and adsorbed amount. BTEX feeding

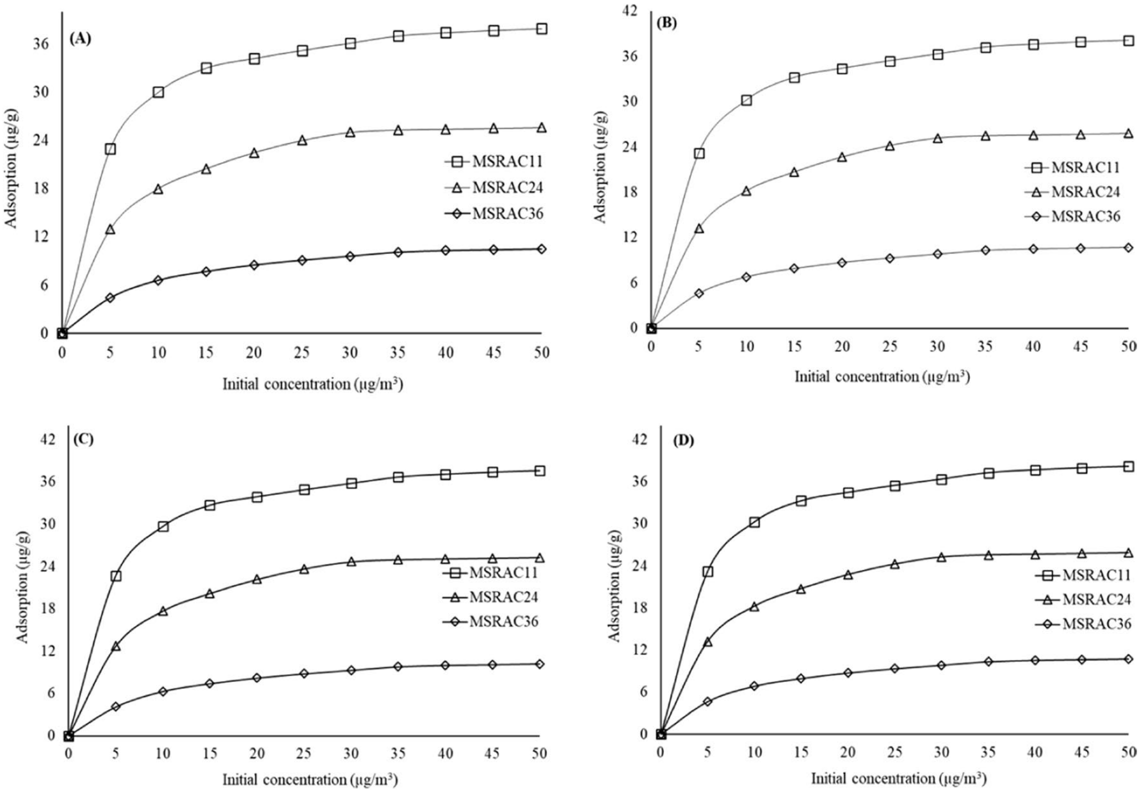

Fig. 3 Adsorption capacities of **A** benzene, **B** toluene, **C** ethylbenzene, and **D** xylene

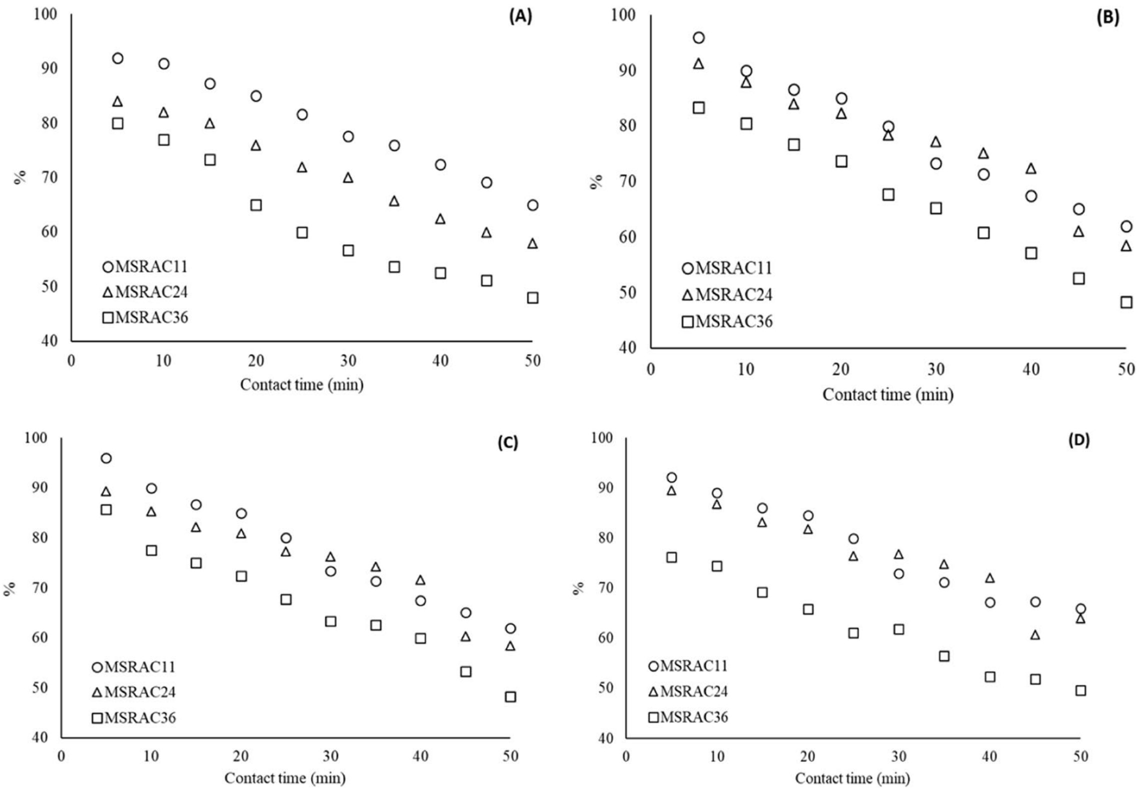

Fig. 4 Removal efficiency (%) of **A** benzene, **B** toluene, **C** ethylbenzene, and **D** xylene

concentration and time were increased until 50 µg/m^3 and 50 min, respectively, and consequently adsorption amount presented a gentle slope. The BTEX adsorption capacity changed according to the process of filling pores, as shown in Fig. 4. The adsorption efficiency (%) decreased as the contact time increased, and even the baseline of adsorption did not reach 50 min. Comparing MSRAC11 and MSRAC24–MSRAC36, it was concluded that a higher initial performance of MSRAC11 led to higher adsorption capacity. The toluene's highest efficiency was obtained in MSRAC11 (i.e., 96%) in the first 5 min, and it was observed that it decreased to 62% as time progressed. The benzene efficiency as the second highest-adsorbing substance was 92%, and the percentage decreased with time and initial concentration.

Figure 4 shows the adsorption properties of each volatile compound against time. The adsorption efficiency (%) is one of the essential parameters which is determined for the non-polar VOCs in such systems. The adsorption efficiency was inversely proportional to contact time. The higher efficiency (%) of BTEX molecules decreases with time which is related with gradual saturation of the sorbents surfaces during the tests.

4 Discussion

Several technologies are exploited to remove BTEX from air streams. However, the success achieved with using adsorption technique is because of its easiness and lower costs, compared to other methods [61–64]. Activated carbons can adsorb BTEX via several mechanisms like π-π interactions, pore filling, and/or hydrogen bonds. In particular, carbon-based adsorbents' production, desorption, and dissemination are carried out as low-cost processes [65]. The amount of BTEX gases in the indoor environment increases under some conditions when the solvents in interior materials such as new furniture, wall paints, and wood coatings are released into the atmosphere at room temperature [66, 67]. Studies have shown that the amount of BTEX in the interior media may harm human health and cell metabolism [68–70]. The success of activated carbons in reducing VOCs to below certain levels has been demonstrated in the literature, and the efficiency has increased despite the variation in BTEX capture and sorption rates of different adsorbents [71]. Lara-Ibeas et al. [72] tried multi-component BTEX adsorption studies with commercial and synthetic adsorbents, namely Carbopack® B (112 m^2/g), SBA-16 (572 m^2/g), and HKUST-1 (1733 m^2/g). The HKUST-1, the adsorbent with the highest surface area, had the lowest BTEX adsorption capacity (11.28 mg/g); however, the SBA-16 adsorbed a total BTEX amount of 30.2 mg/g. Lee et al. [73] tried to synthesize nanoparticles which were finally used to investigate the adsorption performance of 10 ppm BTEX by MIL-100 (Fe) metal-organic framework filter (PMF). The recyclable PMF was placed in a 40 × 20 × 20 cm^3 acrylic testbed, and its performance was found to be between 75.95 and 94.25% for BTEX removal efficiencies. Megias-Sayago et al. [74] successfully synthesized several ZSM-5 to remove gas-phase BTEX. The BET surface area of ZSM-5s obtained ranged from 348 to 386 m^2/g, and they were used to remove a ratio of 1% or 5% of the BTEX initial concentration. Generally, the zeolites can be selective for BTEX, excluding m and o-xylene. Similarly, in this study, research showed that MSRAC11 has higher BTEX adsorption performance due to its micropores volume. It may explain the high BTEX adsorption capacity of MSRAC11 compared to other adsorbents with wider mesopores. However, it is hard to describe behavior with limited studies on adsorption between environmental conditions and concentrations, so more research is needed for a suitable adsorbent with carbon materials [75–79]. Overall, in this work, the BTEX removal experiments showed that adsorption process using activated carbon derived from stems of *Corylus colurna* has an effective performance for BTEX removal. In fact, BTEX molecules significantly adsorb on such an adsorbent via different interactions. Anyway, adsorption of BTEX compounds from environment is still a current research topic and will continue to excite researchers.

5 Conclusion

VOCs adsorption has been an interesting scientific topic over the years due to its health effect significance. Adsorption has been recommended as a process to reduce VOC level in indoor areas. Adsorbents can be produced from various materials and the original materials used for adsorbent preparation usually have high effect on capacity characteristic properties. Numerous low-cost adsorbents from agricultural or lignocellulosic biomass waste have been synthesized and characterized in recent past. In this work, activated carbons (MSRACs) were produced from the stems of *Corylus colurna* by chemical activation with H_2SO_4, H_3PO_4, and HCl, and the adsorbents were used for removal of VOCs from indoor streams. Among the most dangerous VOCs, the BTEX is frequently applied to removal studies and has been successfully carried out with carbon-based adsorbents. Due to the widespread BTEX usage in industrial applications, it is first released to the indoor environment, and the reactions occur. In this article, MSRACs effectively attempted as carbon-based adsorbents in reducing BTEX concentrations. The different behavior of each BTEX compound on the adsorbent depends on the characteristic features of adsorbent. However, some environmental factors, such as humidity, ventilation, and temperature, cause differences in BTEX interactions with the adsorbents, which were optimized. Hereby, the adsorption capacities of each compound were highly relevant to adsorbate-adsorbent interactions and micropores' existence. For future development, the MSRAC11 can be applied to homes, offices, automobiles, and libraries because it has excellent stability and durability for BTEX removal.

Author contributions Kaan Isinkaralar: investigation, data curation, original draft preparation, writing—reviewing and editing. Saule M. Nurmakova: writing—reviewing and editing.

Data availability The data is provided by corresponding author under reasonable requests.

Declarations

Ethics approval and consent to participate Not applicable.

Consent to publish Not applicable.

Competing interests The authors declare no competing interests.

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