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ORIGINAL ARTICLE

Optimal preparation of low-cost activated carbon: the enhanced mechanism of interaction and performance studies for volatile organic compounds (VOCs) capture

Kaan Isinkaralar[1]

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Abstract

The activated carbons developed using *Phaseolus vulgaris* L. (PVACs) were produced to enhance the benzene, toluene, ethylbenzene, and xylene (BTEX) adsorption. The PVACs chemically produced via phosphoric acid (H_3PO_4) from biomass waste and effectively found the adsorption capacity for multi-component BTEX. In accordance with the experiment protocols, a set of systematic operating parameters (temperature, residence time, and initial BTEX concentration) carried out to investigate the mechanism of multi-component BTEX adsorption on the PVACs (PVAC650, PVAC750, and PVAC850 named as carbonization temperature from 650 to 850 °C). The ambient temperature was kept constant at 22 °C, and the differences of BTEX adsorbed amount were clearly demonstrated in the experiments. The results indicate that (1) specific surface area is important for novel application in multi-component BTEX adsorption, and the surface area of PVAC650, PVAC750, and PVAC850 were 881, 1109, and 1007 mg/g; and (2) temperature and relative humidity have an important effect on the absolute BTEX adsorption, which BTEX adsorbed amount decreases at higher temperature and relative humidity. Under the condition of 15% relative humidity (RH), the maximum BTEX adsorption amounts for PVAC650, PVAC750, and PVAC850 were 189, 281, 217 mg/g at, respectively. The average multi-component of BTEX adsorption on PVAC750 is 235 mg/g, indicating that the dominant adsorption process of BTEX at room conditions may be physical adsorption.

Keywords Activated carbon · Biomass pyrolysis · Synthesis techniques · Environmental applications · Organic component

1 Introduction

Clean air is essential for life, and different amounts of pollutants in ambient air limit air quality. Volatile organic compounds (VOCs) are the most important and dangerous several components of air emissions as an essential precursors for tropospheric ozone and secondary organic aerosols [1, 2]. Benzene, toluene, ethylbenzene, and xylene (BTEX) are the most toxicities and reactivities substances among VOCs that profoundly influencing human health and the ecological environment [3, 4]. Ninety percent of people worldwide are exposed to substandard environmental concentrations of BTEX in indoor environments due to indoor VOC often exceeds the outdoor concentrations [5]. BTEX, a component of poor air quality and decreased climate conditions, poses a public health hazard as a result of exposure in environmental and occupational settings [6–8]. As a result of the widespread use of BTEX-containing solvents consumed as a result of the widespread use of various manufacturers, very high amounts are released into the atmosphere [9–11]. Despite this, the majority of them are released to the environment in significant quantities in recycling processes, which can allow the contamination.

Considering the environmental protection laws are swiftly put into effect in order to use and disseminate techniques to reduce environmental BTEX releases [12–14]. Environmental laws and regulations used for this purpose emphasize the use of source reduction techniques, if possible, and emission reduction techniques, if not possible [15]. Although BTEX levels in the atmosphere are predicted to increase according to the annual reports of various environmental agencies, it is noted that countries are not taking enough precautions [16, 17]. The search for methods

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✉ Kaan Isinkaralar
kisinkaralar@kastamonu.edu.tr

[1] Department of Environmental Engineering, Faculty of Engineering and Architecture, Kastamonu University, 37150 Kastamonu, Türkiye

to mitigate the emerging risks has become a popular topic [18–20]. Recovery methods have been made to develop efficient VOC abatement technologies include membrane separation [21], absorption [22], adsorption [23], condensation [24]; but catalytic oxidation [25], biodegradation [26], thermal oxidation [27] and plasma catalysis [28] have also been favored as disposal methods. However, adsorption using carbon materials as VOC adsorbents is recognized as one of the most cost-effective and potentially promising management strategies [29–32].

The worldwide importance of recovery (in terms of sustainable environment) has been better understood, and adsorption has been recognized by the scientific community as one of the successful methods used [33]. It has been a matter of curiosity for the purpose of treatment or purification of BTEX gases. The investigation of the process in which gas molecules are attracted by physical bonds as a surface phenomenon has become a very common and current phenomenon [34–36]. The surface structure and physico-chemical properties of the adsorbents used are considered as the vital organ of the process [37]. It is known that the use of various adsorbents directly affects the efficiency [38, 39]. The basic parameters of the material used such as form, porous environment, and the higher micropore volume lead to increased adsorption uptake capability [40]. In addition to adsorbents with small pore size, reasonable porosity, high micropore volume, the media properties that allow the molecules desired to be held on the surface to reach the interior of the adsorbent have also gained importance [41]. Adsorption materials are the main factor directly affecting the investment cost, operating cost, and safety in VOC adsorption technology [42–44]. Successful adsorption process is formed by differentiating the conditions that help adsorption to take place [45]. In addition, adsorption capacity, kinetics, and equilibrium conditions should be examined for a better understanding of the adsorption process. For successful adsorption in multicomponent BTEX studies, it is very significant to know the adsorbent with micropore structure and the concentrations of the components present in the system [46]. Various biomaterials have been evaluated as biosorbents for the removal of gas phase pollutants. The most commonly used are lignin-based species, agricultural wastes, and economically obsolete species found in forests. Recently, crude and upgraded or modified forms of biomass have been investigated more thoroughly for this purpose due to their low cost and their potential to adsorb a wide range of gas phase BTEX, are sustainable, replenishable, renewable and highly abundant natural resources, and demonstrate good resilience and productivity. In some studies, specific feedstocks (a solid waste from various agricultural wastes, woody biomass, and sewage sludge, etc.) tea residue [47], hybrid willow [48], ashitaba [49], walnut shells [50], pineapple [51], pomelo peel [52], coconut shells [53], corncob [54], castor capsule beans [55], tea leaf [56], peanut shells [57], pitaya peels [58], horse chestnut shell [59], mangosteen peel [60], slash pine [61], rice hulls, and wood-based [62, 63] are utilized. The utilization of all these adsorbents for VOCs removal and purification using crude and modified carbon-based activated carbons was investigated in environmental applications [64, 65]. The waste biomass production is widespread worldwide, and it is known that after threshing, approximately 60% of the whole plant mass gets into manufacturing residues [66]. It is therefore of great importance to provide solutions to reuse, recycle, and reduce Anasazi beans waste which consists of bean pods, stems, and leaves. Because of the presence of specific molecular groups, these lignocellulosic substances have good performance and high potential for the elimination of diverse gases [67].

The aim of this paper can be stated as follows: (i) to produce the mechanism behind pore formation in activated carbon produced from Anasazi beans waste, (ii) to investigate the usability of BTEX by a more economical and environmentally friendly adsorption, and (iii) investigation of factors affecting BTEX adsorption such as contact time, adsorbent dosage, and initial multi-component BTEX concentration.

2 Experimental section

2.1 Materials

Phosphoric acid (H_3PO_4), potassium hydroxide (KOH), and BTEX (benzene, toluene, ethylbenzene, and xylene) standard solution were purchased from Sigma Aldrich, USA. Throughout the experiments, all reactants and N_2 were of chemical grade as 99% purity and deionized water from Milli Q plus, Merck Millipore Germany. Once opened, the chemicals used hidden in vials with glass stoppers and in the dark at 4 °C. Standard stocks were dissolved in methanol and eluted to provide a set of standards for sampling.

2.2 Preparation of activated carbon

Anasazi beans (*Phaseolus vulgaris* L.) peels (PVP) were collected from local producer as precursor for this study due to its wide availability in Kastamonu, Türkiye. *P. vulgaris* was initially small-sized and was repeatedly cleaned many times with boiling distilled water to eliminate impurities. Then it was dried at 80 °C for a week before going on to the following steps. The pyrolysis of the biomass was conducted by operating a fixed bed reactor. The chemical activation procedure was carried out in the second step by impregnation of PVP with concentrated H_3PO_4 (85% wt) at 150 °C at the optimum incorporation ratio (50 mL H_3PO_4 per 20 g PVP as predetermined the optimum ratio). This was followed by a heating process at 100 °C for 24 h of dwell time in an oven. The precursor of dried PVP was filled into a graphite crucible and pyrolyzed in a muffle furnace at three different temperatures (650 °C, 750 °C, and 850 °C) for 2 h and held for 3 h under continuous flowing nitrogen (99.99%) at a flow rate of 100 mL/min. The black colored material was spilled into deionized water, and the pH was regulated to 7.00 using 1.00 N KOH. Next, it was recovered after filtration and drying at 105 °C. Also, it was ground into a fine powder (size of ≤ 250 µm) and denominated as PVP-derived activated carbon (PVAC650, PVAC750, and PVAC850).

2.3 Analytical methods and characterizations

Both bulk PVP and prepared PVACs were characterized using a QuantaTM 250 FEG scanning electron microscope (SEM) to determine the morphological features of the samples at 5000 × enlargement. The Brunauer–Emmett–Teller (BET) and density functional theory (DFT) method to evaluate surface properties and porosity through chemisorption-physisorption analyzer model Autosorb-1-C (Quantachrome Instruments). The elemental compositions of the raw PVP and PVACs were detected by EuroVector, EA3000 elemental analyzer.

2.4 Design of BTEX adsorption

The batch adsorption operation was designed to investigate the adsorption of BTEX vapor by PVACs (0.1 g of each adsorbent per experiment) as adsorbent. The temperature of the reactor where the adsorption process would take place was kept constant in a controlled manner at predetermined temperatures of 20 and 30 °C, respectively. In addition, in order to determine influence of exposure period (110 min), samples were pre-prepared with an initial BTEX concentration of 350 mg/m^3 in the system with a vacuum pump (ratio of 70 mL/min) by AirLite 110–100, SKC at different times. Tenax®-TA single sorbent tubes were used to determine the concentration of BTEX in the reactor by Thermo Scientific Trace 1300 gas chromatography/mass spectrometry detector (GC–MS) and ISQ-QD Thermo Fisher Scientific. The breakthrough pattern was quantified with a Markes Unity Series 2 thermal desorber combined with GC–MS. TG-624 capillary columns were employed to quantify BTEX according to US EPA TO-17 Method [68]. Equation (1) were adopted to estimate the multi-component BTEX equilibrium adsorption capacity.

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

where C_i (mg/L) and C_o (mg/L) represents inlet and outlet recorded amount of BTEX at equilibrium condition. Also, system feed flow rate (F) of 150 mL/min and 0.2 g of PVACs (W) were performed for selected sorption experiments.

3 Results and discussion

3.1 Characteristics of the PVACs

The data of proximate and elemental contents for PVP, PVAC650, PVAC750, and PVAC850 are plotted in Fig. 1. It is clear that the percentage of fixed carbon content is higher in PVACs than in PVP, respectively. PVP for fixed carbon, volatile matter, moisture, and ash contents were detected as 14.55, 69.45, 2.10, and 13.9%, respectively, whereas for PVAC650, PVAC750, and PVAC850, fixed carbon, volatile matter, moisture, and ash contents were 66.2, 69.4, and 69.5%; 28.05, 24, 22.7%; 3.2, 2.8, and 1.7%;

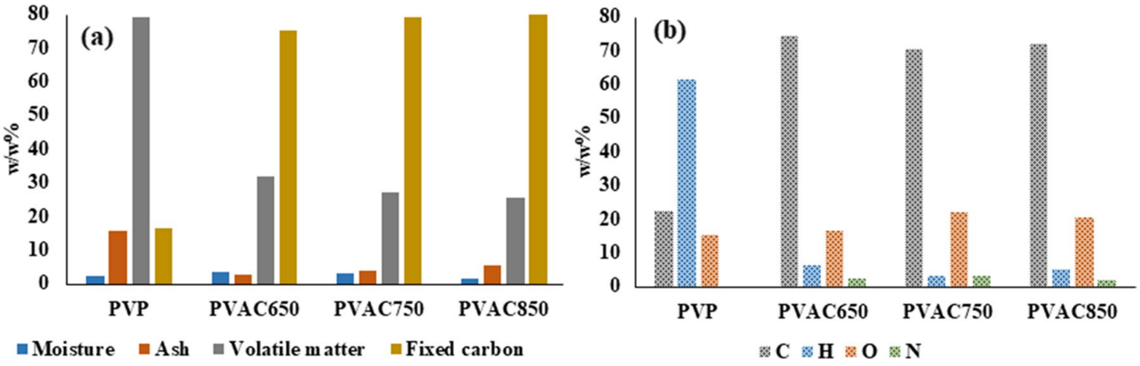

Fig. 1 **a** Proximate properties (w/w%) and **b** ultimate composition (w/w%) of PVP and PVACs

2.55, 3.8, and 5.1%. The proximate amount of PVR is lower than PVAC650, PVAC750, and PVAC850. PVR has high volatile matter which is reduced from 69.45 to 22.7%. This reduction was due to carbonization and activation processes at high temperatures. The fixed carbon increased remarkably from 14.55 to 69.5% due to favorable physiochemical activations in their composition. Despite this, the decline in ash content suggested that PVAC650, PVAC750, and PVAC850 are promoting adsorbents for BTEX remediation. The elemental content appeared with a trend comparable to the proximate analysis. In addition, percentage of carbon (C) element in the precursor PVR increased from 22.8 to 74.5%, considering appropriate capacity and effectiveness for BTEX purification.

Notable inputs of hemicellulose, cellulose, and lignin to the weight and properties of porous PVACs. The properties of lignocellulosic compositions of PVP were demonstrated that the composition of the weight percentages of hemicellulose, cellulose, and lignin were 25.1, 47.5, and 27.4%, respectively. The broad specific surface area and porosity surface remain critical characteristics that encourage the exceptional capability and efficacy of PVAC650, PVAC750, and PVAC850. This improvement in the mesoporous surface area, the BET surface area, the average pore diameter, and the total pore diameter is shown in Fig. 2. PVAC650, PVAC750, and PVAC850 demonstrate the enhanced BET surface areas of 881, 1109, and 1007 m^2/g and total pore volumes of 0.78, 0.71, and 0.73 cm^3/g. The significant increase in total pore volume and BET surface area was due to the flow of volatiles and water from the PVR. The PVAC650, PVAC750, and PVAC850 exposed an average pore diameter of 2.17, 1.43, and 1.67 nm, respectively.

Scanning electron micrographs of PVR, PVAC650, PVAC750, and PVAC850 are given in Fig. 3. They were prepared under optimized conditions where the surface morphologies contained visible voids, slits, pores, and holes of various sizes. The combination with H_3PO_4 and carbon also evolves, creating highly advanced pores. The PVR precursor is prone to the rise of irregular shapes and the complete absence of porosity in nature. Figure 3b shows that PVAC650 has a large number of smooth and homogeneous pores on its surfaces. In Fig. 3b and c, PVAC750 and PVAC850 developed fewer but homogeneously immobilized pores than PVAC650 due to evaporation of H_3PO_4 during carbonization. Therefore, the verification of the effectiveness of chemical activation in combination with H_3PO_4 heating at 750 °C in the preparation of a precisely designed and integrated composition of PVAC750 shows that activation using H_3PO_4 and the carbonization process at 750 °C form expanded aggregates and developed pore spaces on PVAC650, PVAC750, and PVAC850.

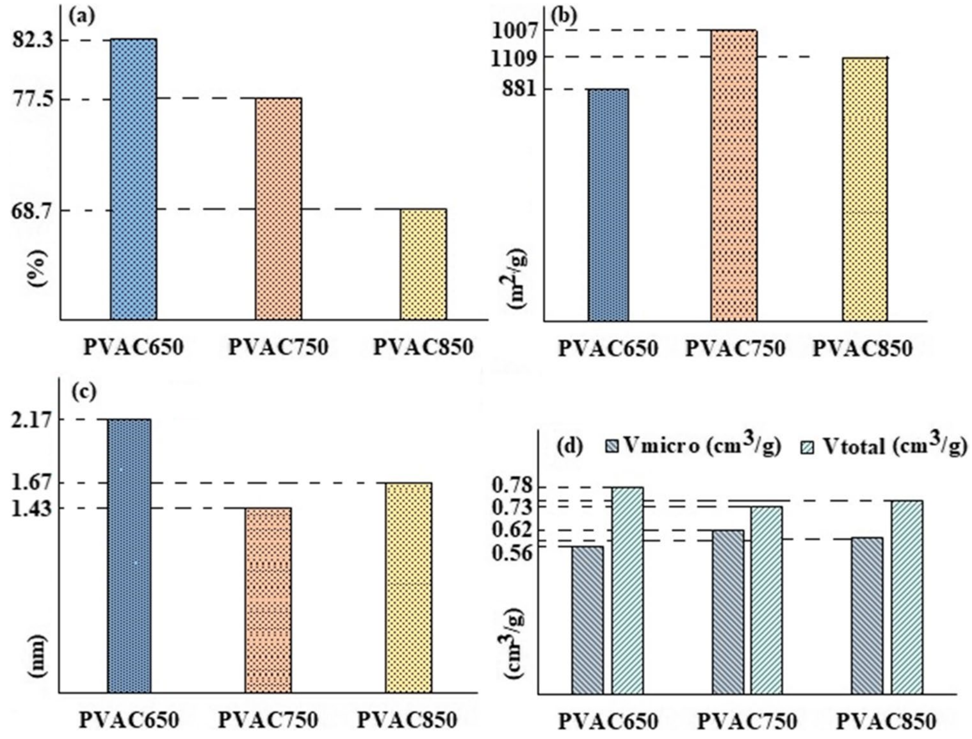

Fig. 2 **a** Yield of production. **b** BET surface area. **c** Mean pore diameter. **d** Total pore and micro volume of PVACs

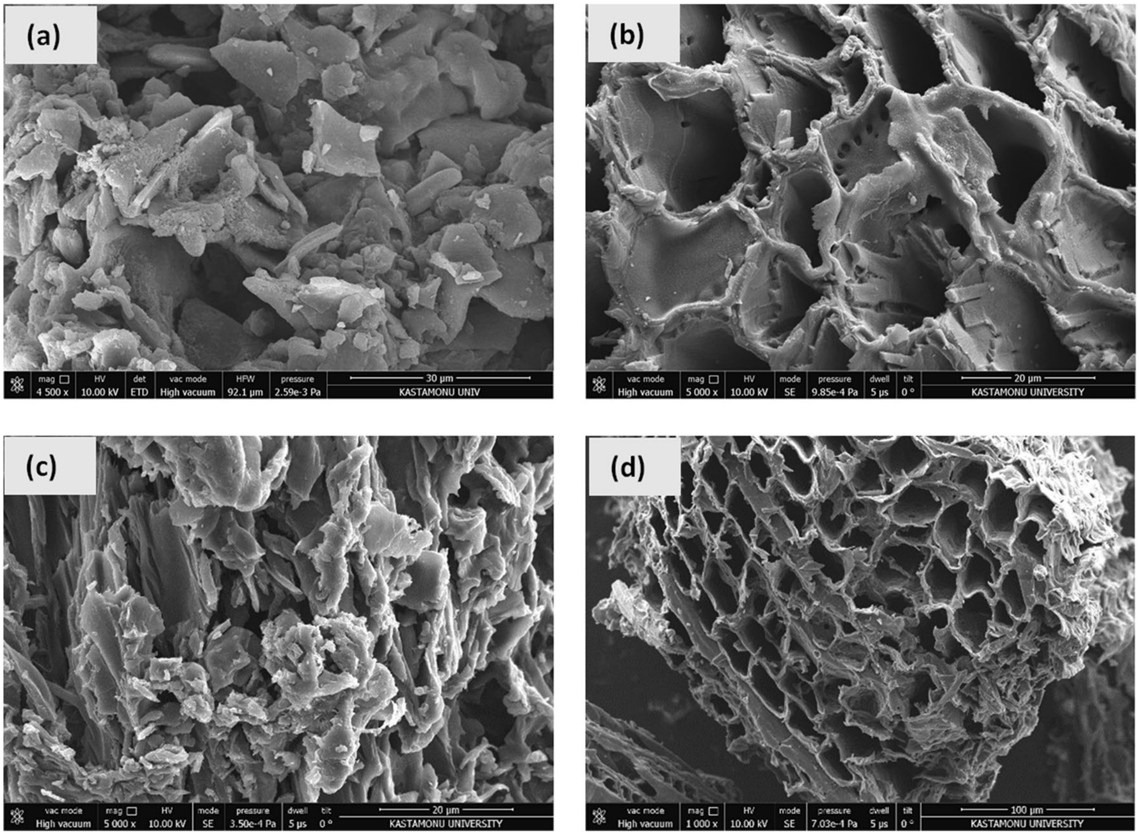

Fig. 3 Scanning electron micrographs of **a** PVP 4500×, **b** PVAC650 5000×, **c**: PVAC750 5000×, and **d** PVAC850 1000×

3.2 BTEX adsorption characteristics of PVACs

The adsorption condition differences brought about a substantial variation in the adsorption capacities in Fig. 4. Especially the alterations in both temperature and humidity reveal differences in equilibrium conditions. Although the adsorbents and amounts used are the similar, the results are considerable. The multi-component BTEX adsorption capacities of PVAC650, PVAC750, and PVAC850 for 65% RH are 173, 247, and 201 mg/m at 20 °C and 113, 172. and 133 mg/m at 30 °C. The results were inversely proportional to the temperature acting on the pores for the total amount of BTEX at 65% RH. Nevertheless, there is a significant decline in the adsorption capacity when the temperature is raised for PVAC650, PVAC750, and PVAC850 from 20 to 30 °C as 53.10, 43.60, and 51.12%. In fact, it is clear from the quantity of BTEX adsorbed that both

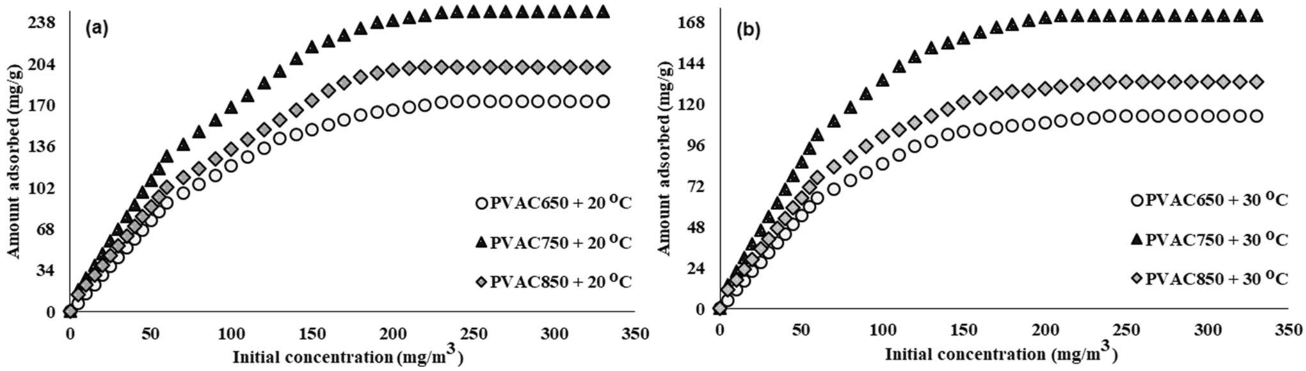

Fig. 4 Adsorption capacities of **a** 20 °C and **b** 30 °C at 65% RH

temperature and humidity have a significant effect on the adsorption capacity of BTEX.

Humidity is a critical factor which was examined in the behavior of the PVAC650, PVAC750, and PVAC850 at 15% RH and 95% RH. The ambient temperature was kept constant at 22 °C, and the differences of BTEX adsorbed amount were clearly demonstrated in the experiments in Fig. 5. BTEX adsorption amounts for PVAC650, PVAC750, and PVAC850 were 189, 281, and 217 mg/g at 15% RH, respectively, while they were 112, 188, and 161 mg/g at 95% RH. Although BTEX adsorption was reached at equilibrium condition in 46 min at 15% RH, it reached equilibrium in 32 min at 95% RH. Increasing humidity percentage in BTEX adsorption capacity for PVAC650, PVAC750, and PVAC850 was recorded with decreasing rate as 68.8, 49.5, and 35.8%, respectively.

4 Discussion

In terms of volatile organic compounds (VOCs) and the breadth of their industrial use, benzene, toluene, ethylbenzene and xylene (BTEX) are four solvent VOCs that are used extensively in industry and at home as chemical intermediates, solvents, fuel additives, and cleaners [69, 70]. They pose serious risks to public health through environmental exposure. Although research on these risks has been adequately investigated, it is still a hot topic [71, 72]. The main reason for this is that the adsorbents used for removal are of economic importance and regeneration is safe. Another problem is the widespread practical use of the produced adsorbents in household, commercial, and industrial products [73]. Considering their benefits, prevalence, and economic importance, the production of activated carbon-based adsorbents is effective in terms of widespread availability of the raw material used, cost/performance, and regeneration [74, 75]. However, BTEX is easily dispersed in air at high vapor pressure and ambient temperatures, which affects adsorption conditions [76]. It rapidly adheres especially to carbon-based adsorbents and is distributed according to the condition of the pores [77].

The physicochemical state of activated carbons is highly concentrated with specific dispersion due to more or less affinity. The main focus of related research on BTEX removal is the efficiency and lifetime of the adsorbent used. Although the removal of BTEX components by adsorbents is widespread, there are currently a limited number of adsorbents that show high efficiency at high concentrations in this multiple removal. The multicomponent BTEX adsorption process is usually characterized by the behavior of a multi-component gas mixture in porous solid media, where the adsorbate molecules depend on the movements of the molecules that manage to enter the micropore volume. The most important condition in the BTEX adsorption process is adsorption equilibrium [78, 79]. In the study by Bhave and Yeleswarapu [80], the adsorption equilibrium of single or multiple BTEX components is important to understand how much of them can fit on the solid adsorbent. The study of adsorption kinetics of single and multicomponent systems is used to understand the importance of multicomponent systems in the formation of adsorption equilibria. Depending on the production models of adsorbents, process and equipment, and changes in ambient conditions (temperature, humidity, and setup), their concentrations can be reduced. In other studies, BTEX control techniques are widely used when the technology used is limited or not available [81, 82].

Although BTEX gases physically interact with the adsorption method, there are cases where they can interact chemically as the process temperature increases. This situation is known to cause difficulties in the separation of BTEXs and causes the desorption process to become difficult.

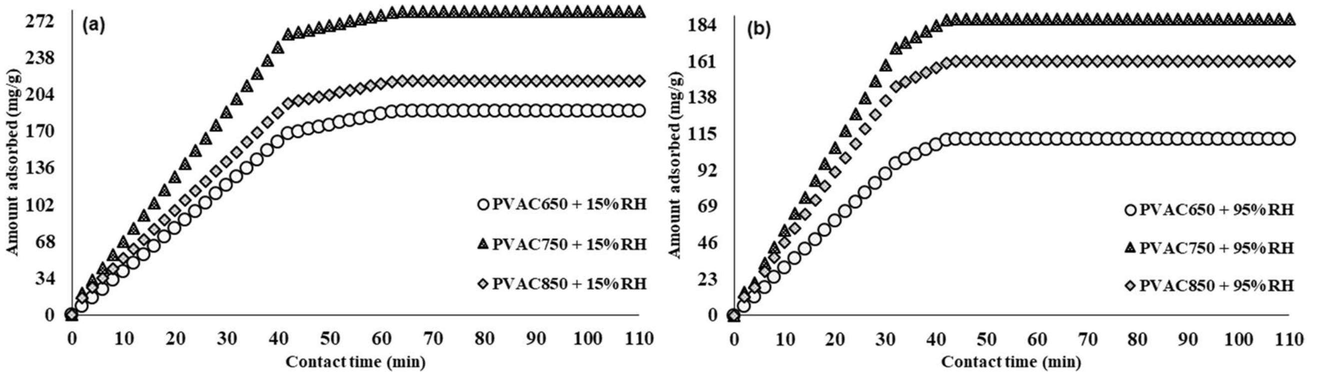

Fig. 5 Contact time of **a** 15% RH and **b** 95% RH at 22 °C

5 Conclusions

Studies on the adsorption mechanisms of volatile organic compounds (VOCs) in the gas phase are of great importance for the accumulation and reserve evaluation of various economically valuable gases that can have toxic effects in the environment. BTEX is typically detected at relatively low concentrations in air urban areas, but regional levels significantly vary by geography, climate, season, weather and closeness to industry, sewage treatment, and transportation. Exposure to BTEX in any community poses a clear risk to public health. While there is empirical evidence of harmful health and neurobehavioral consequences of exposure to individual components of BTEX, these effects are usually mitigated by active or passive removal methods. The most effective solution used is lignin-based activated carbon. Gas adsorption technology is considered an effective and economical control strategy due to its potential to recover and reuse both adsorption material and VOCs. Increasing temperature and relative humidity leads to a lower multi-component BTEX adsorption capacity, which makes it more difficult to adsorb multi-component BTEX molecules. Researchers will greatly benefit from this review work as it will provide a comprehensive understanding of the preparation of activated carbon produced from agricultural and forest biomass as waste material, different synthesis parameters, new synthesis strategies, and the significant success in the removal of multi-gaseous BTEX for environmental remediation. Furthermore, these results confirmed that the use of carbon-based activated carbons in other toxic gas treatment applications can be of interest to researchers working in the same field.

Author contribution This study is written entirely by Kaan Isinkaralar.

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

Declarations

Ethics approval Not applicable.

Competing interests The authors declare no competing interests.

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