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ACTIVATED CARBON ADSORPTION BEHAVIOUR OF TOLUENE AT VARIOUS TEMPERATURES AND RELATIVE HUMIDITY

Filtration plays a major role in managing the rapid increase of environmental pollution and deterioration of atmospheric air quality. One of the most damaging noxious gases in the atmosphere is toluene, which is a common gas that appears in crude oil and is used in various production processes. The adsorption behaviour of activated carbon was examined in dependence of the concentration, temperature and relative humidity. The results showed that increasing air temperature decreased the time to reach the maximum saturation ratio, which occurred due to the increasing pore diffusion coefficient and pore diffusion rate.

1. INTRODUCTION

The environment suffers from pollution on a daily basis: industrialisation paved the way for pollution, especially in the 19th and 20th centuries [1]. The increase in the worldwide population, deforestation and burning of fossil fuels pose a danger to human health. Human beings have significantly intensified worldwide pollution and global warming. Greenhouse gases such as VOCs (toluene, benzene, *n*-hexane and isopropylene), CO_2 , SO_2 and NO_x , which are detrimental to human health, are released into the atmosphere during combustion processes. In addition, VOCs are generally found in solvents for varnishes, resins, oils, paints, cleaners and adhesives, and have adverse effects on human health [2].

Toluene is one of the most common compounds in indoor air, the concentration of which was measured as $87 \ \mu g/m^3$ in Izmir, Turkey [3]. The neurotoxic effects of toluene have been examined via animal experiments. The animals' learning and mental capacity

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were negatively influenced after exposure to a toluene concentration of 80 ppm [4]. Forty two people volunteered for a clinical short-term study in which a toluene concentration of 75 ppm led to headaches and other physical and mental aberrations [5]. The maximum recommended indoor toluene concentration is 4 ppm for short-term exposure and 0.6 ppm for long-term exposure [6]. The annual mean concentrations of toluene in different areas of Germany from 1989 to 2014 are given in Fig. 1.



Fig. 1. Annual mean concentrations of toluene in various areas of Germany from 1989 to 2014 [7]

The amount of toluene from traffic in Germany tended to decrease until 2007. After that year, the amount of toluene again increased from year to year. In rural areas, toluene was not absent in the air until 1989, and a limited amount of toluene was measured in rural regions thereafter.

There are various filters to dispose of particulate matter, such as ionic, high-efficiency particulate air (HEPA), carbon air and ultraviolet light air ones [8]. Carbon air filters are activated after being treated with oxygen and are suitable for adsorbing chemicals and noxious gases, such as toluene or benzene. The activated carbon air filters have small pores, which make them more convenient for filtering and trapping pollutants. In recent years, in order to improve the filtering capacities of the activated carbon filters, great importance has been attached to the modification of active carbon. There are many methods for modifying activated carbon to improve surface shape and adsorption capacities of the filters. Several of the most common modification methods include plasma treatment, acid treatment, impregnation treatment and base treatment, which involve the chemical treatment of active carbon at various temperatures and time durations [9]. Okoniwaeska et al. [10] investigated the adsorption behaviour of varous toxics gases with modified activated carbon. The results showed that the modified activated carbon has a positive effect on the removal of gases. Cao et al. [11] studied the preparation of low-cost activated carbon by ZnCl₂ activation. The result showed that the activation temperature of 550 °C and activation time of 60 min were the optimal conditions to adsorb methylene (MB) and methyl orange (MO).

The behaviour of toluene adsorption on activated carbon under atmospheric conditions (temperature, toluene concentration and humidity) is just as important as the modification of the activated carbon. There are many studies on the adsorption of toluene. Heinen et al. [12] compared the adsorption capability of water and toluene on modified activated carbon filters. Cheng [13] described the VOC adsorption on granular activated carbon for various inlet concentrations of toluene (102-2652 ppm) at 27 °C and relative humidity of 72±3%. Martinez de Yuso et al. [14] analysed the adsorption and recovery behaviour of almond shell-derived activated carbon filters for low concentrations of two VOCs (n-hexane and toluene). The high adsorption capacity of n-hexane and toluene was obtained at low concentration (253 mg toluene/g and 122 mg *n*-hexane/g). Shiue et al. [15] described the vapour adsorption characteristics of toluene on activated carbon. To determine the capacity and adsorption efficiency, the testing temperature and relative humidity were maintained at 28±1 °C and 40±2%, respectively. Lorimier et al. [16] studied activated carbon cloths and felts for indoor toluene removal under toluene contents between 21 and 18 160 mg/m3. Cheng et al. [17] found a fitting model for toluene adsorption with the values of parameters K and W_0 of 1.1×10^{-9} and 57.73 kg/m³, respectively.

There are limited papers on the adsorption characteristics of toluene, but only several of these focused on the adsorption of toluene onto activated carbon. Few authors investigated the effect of relative humidity on the adsorption of VOCs to date. Furthermore, most of the studies involve adsorption of toluene under high concentration rates; however, a vast majority of those papers analysed the adsorption of toluene on catalysed activated carbon.

In this study, breakthrough curves for toluene were examined to reveal its adsorption characteristics. The adsorption curve was drawn to compare the time to reach the maximum saturation ratio under various working conditions.

2. EXPERIMENTAL METHOD

Testing system. Toluene gas was supplied to the experimental setup as a toxic gas. This study was performed according to the DIN 71460 standard, which describes the cabin air filtration standard and the test methods for measuring the dynamic gas adsorption of air filters [18]. A schematic view of the experimental setup is shown in Fig. 2.

The atmospheric air is drawn through a fan and is cleaned of pollutants and noxious gases via high efficiency particulate air (HEPA) and activated carbon filters. The next component of the filter test rig contains different sub-assemblies such as an air heater, an air cooler and a steam humidifier. Between the air heater and the steam humidifier, there is a ventilator with a volume flow rate capacity between 60 and 700 m³/h; throughout the experiment, the air velocity is set at 0.2 m/s. The air heater has a rated power of

9 kW. The air cooling loop is driven by cold water at 7 °C. To maintain a constant temperature throughout the procedure, a cold water tank 0.95 m³ in volume is implemented, enabling variation in the rated experimental parameters to be reduced to 0.3 °C. The water vapour humidifying the air stream is supplied by the steam humidifier. The steam is produced in a steam generator in which the spray rate is controlled by a test channel using a steam distribution pipe. The hourly steam capacity of the steam humidifier is 10 kg/h. These sub-assemblies are intended to condition the air flow with regards to temperature and relative humidity in the experimental setup. The temperature can vary from 15 °C up to 33 °C, and the relative humidity can be adjusted up to 90%. The temperature, relative humidity, air speed, and toluene concentration in raw and treated air taken from retrieved samples are transmitted to the system control unit. Afterwards, the values are transferred from the system control unit to the computer system. Those values are recorded in an interval of 0.067 min.



Fig. 2. Schematic view of the experimental setup

A TMP36 measurement sensor was used to monitor temperature, humidity and air flow rate. After measuring the air parameters, toluene gas was supplied via the mass flow controller to the cleaned atmospheric air in the gas supplier section. A corresponding flow regulator at the inlet of the activated carbon filter section ensured homogeneous distribution of the air–gas mixture. After the flow regulator, a round cylindrical bed was mounted in which a certain amount of activated carbon should be homogeneously distributed. The fixed bed method was used in this experiment. Measuring probes were installed just before and after the activated carbon filter in order to measure the toluene concentration in the raw (C1) and treated air (2). The measurement process was accomplished by two flame ionisation detectors (FID).

After the preparation of the experimental setup, the sub-assemblies were calibrated, and the experiment conditions (humidity and temperature) were later adjusted. In the next step, the activated carbon filter was inserted into the test rig. Meanwhile, the toluene gas was injected into the air. Before and after the activated carbon filter, the toluene gas concentration in the air was recorded at predetermined measurement time intervals. The experiment continued until the C2/C1 ratio reached the breakthrough point. The test procedure was repeated for various temperatures, humidity ratios and concentrations.

Activated carbon. Coconut shell-based activated carbon was selected for our experiments. The activated carbon used in this research was supplied by Trabac Co. in Vietnam. Images of the activated carbon used in the experiment (a) and its pore structure various magnifications are shown in Fig. 3.



Fig. 3. Photographs of the activated carbon bed used in the experiment (a), and its pore view at a scale of 10 μ m (b) and 1 μ m (c) (SEM)

The coconut shell-based activated carbon was heated up to 75 $^{\circ}$ C in a furnace to vaporise its moisture over the course of 1 h. Next, the dried activated carbon was scattered on the surface of the activated carbon bed to a thickness of approximately 1 mm. The characteristics of the activated carbon and its structural properties are given in Table 1.

Table 1

Parameter	Value
Pore volume, cm ³ /g	0.43
Ash content (dry basis), %	3.2
Grain density, kg/m ³	2100
Grain size, mm	0.42-0.85
Particle size, mm	0.250-0.595
Amount of activated carbon used, g	0.79
Thickness, mm	1
Bed surface area, cm ²	19.15
Weight per surface	400

Characteristics of the coconut shell based activated carbon and structural properties of the bed

Adsorption process on activated carbon. The schematic representation of adsorption process on activated carbon is given in Fig. 4.



Fig. 4. Schematic representation of adsorption process on activated carbon

The raw gas, being a mixture of air and toluene (adsorbate), was passed through the activated carbon (adsorbent) at a constant air temperature, raw gas concentration and relative humidity in each experiment. During the experiment, the gas concentration of before (C1) and after (C2) contact with active carbon was recorded. The point at which the C2/C1 (saturation ratio) reached 100% was identified as the saturation point.

The DIN 71460 Part 2 standard defines the test procedure of the filters for gas adsorption. In this standard, the temperature, relative humidity and gas concentration are defined as 23 °C, 50% and 80 ppm. The experiments were carried out with different toluene concentrations of 4, 8, 10, 20, 40 and 80 ppm.

3. RESULTS AND DISCUSSION

The results of the experiments are shown in Figs. 1–16 as the time dependences of the C2/C1 concentration rates representing concentrations of toluene in air before (C1) and after (C2) adsorption. The figures contain curves for varying relative humidity at a constant toluene concentration and temperature. The saturation ratio curves for 4 ppm and 8 ppm of toluene at T = 33 °C, $\phi = 50\%$ are shown in Fig. 5.



Fig. 5. Saturation ratio curves for 4 ppm and 8 ppm of toluene at T = 33 °C, $\phi = 50\%$

Activated carbon reaches the maximum saturation ratio more slowly at 4 ppm than at 8 ppm at T = 33 °C, $\phi = 50\%$. The maximum saturation ratio was evaluated after 700 min as 80% for 4 ppm, and at 460 min as 90% for 8 ppm of toluene concentration. The increase of the curve slope in the figure is directly related to adsorption. Therefore, the time to reach the maximum saturation ratio of the active carbon is shorter at 8 ppm than at 4 ppm. Moreover, the increase in toluene concentration in raw air decreases the time needed to reach the saturation point. The saturation ratio curves for 4 ppm and 8 ppm of toluene at T = 33 °C, $\phi = 70\%$ are shown in Fig. 6.



Fig. 6. Saturation ratio curves for 4 ppm and 8 ppm of toluene at $T = 33 \text{ °C}, \phi = 70\%$

At 8 ppm of toluene concentration, the activated carbon arrives at 95% of the saturation ratio after approximately 300 min. However, for the 4 ppm toluene concentration, the activated carbon reaches its maximum saturation ratio of nearly 88% after 540 min. When both the 4 ppm and 8 ppm curves are investigated together at 33 °C and 70% relative humidity, it is clearly seen that the adsorption of the toluene increases along with the concentration. Accordingly, the time needed to reach the maximum saturation ratio decreases with the increase of the toluene concentration in raw air. The saturation ratio curves for 4 ppm and 8 ppm of toluene gas concentration at T = 33 °C, $\phi = 90\%$ are shown in Fig. 7.



Fig. 7. Saturation ratio curves for 4 ppm and 8 ppm of toluene at T = 33 °C, $\phi = 90\%$

The maximum saturation ratio and the time to reach the saturation ratio are found as 90% and 450 min for the 4 ppm and 95% and 250 min for the 8 ppm of toluene in raw air, respectively. Taken together with the previous three diagrams of low concentrations of toluene gas (4 ppm and 8 ppm), the time to reach the maximum saturation ratio is shorter with increasing relative humidity at a constant temperature (T = 33 °C). The adsorption rate of toluene increases upon increasing concentration. However, as is seen in Figs. 5–7, by considering the 50%, 70% and 90% relative humidities together, the slopes of the saturation ratio curves increase slightly with the increase of the humidity.

Shiue et al. [15] studied the adsorption rate of toluene at velocity of 0.152 m/s, adsorbent mass of 1.75 g, relative humidity of 40%, an air temperature of 28 °C and toluene concentration in raw air of 10 ppm. Results indicated that the time to reach the maximum saturation ratio was 330 min, which is close to the result obtained in the present study at a relative humidity 50% and raw air toluene concentration of 8 ppm. Many experiments performed by Mohan et al. [19] focused on the adsorption rate of toluene in raw air at low concentrations (5 ppm, 10 ppm and 15 ppm), and indicated that the time to reach the maximum saturation ratio decreased upon increasing concentrations. Our experiments at 4 ppm and 8 ppm showed the same trends as this study. Moreover, the adsorption of toluene on activated carbon was also examined for higher toluene concentrations in raw air (20 ppm, 40 ppm and 80 ppm). The saturation ratio curves for 20 ppm, 40 ppm and 80 ppm of toluene at T = 15 °C, $\phi = 50\%$ are shown in Fig. 8.



Fig. 8. Saturation ratio curves for 20 ppm, 40 ppm and 80 ppm of toluene at T = 15 °C, $\phi = 50\%$

At T = 15 °C and $\phi = 50\%$, the activated carbon reaches its maximum saturation ratio in a shorter time at 80 ppm of toluene concentration than at 20 ppm and 40 ppm. The maximum saturation ratio and the time required to reach it for 20 ppm, 40 ppm and 80 ppm of the toluene in raw air was found to be 92% at 205 min, 95% at 110 min and 95% at 80 min, respectively. The saturation ratio curves for all concentrations of toluene at T = 15 °C, $\phi = 70\%$ are shown in Fig. 9.



Fig. 9. Saturation ratio curves for 20 ppm, 40 ppm and 80 ppm of toluene at T = 15 °C, $\phi = 70\%$

Activated carbon arrives at its maximum saturation ratio in a shorter time at 80 ppm of toluene than at 40 ppm. The maximum saturation ratio and the time needed to reach it for 20 ppm, 40 ppm and 80 ppm is calculated as 88% 210 min, 95% 120 min and 98% 90 min, respectively. The saturation ratio curves for 20 ppm, 40 ppm and 80 ppm of toluene at T = 15 °C, $\phi = 90\%$ are shown in Fig. 10.



Fig. 10. Saturation ratio curves for 20 ppm, 40 ppm and 80 ppm of toluene at T = 15 °C, $\phi = 90\%$

The maximum saturation ratio and the time required to reach it for 20 ppm, 40 ppm and 80 ppm of toluene in raw air was found as 90% at 210 min, 95% at 130 min and 98% at 110 min, respectively. The time needed to reach the maximum saturation ratio increased upon increasing relative humidity at a constant temperature (T = 15 °C), while the adsorption rate of the toluene increased upon its increasing concentration (Figs. 8–10). The time needed to reach the breakthrough point decreased upon increasing concentration. However, when the breakthrough curves are examined according to the changing relative humidity along with constant temperature and concentration, the adsorption rate decreased and the time to reach the maximum saturation ratio increased for the concentrations of 20 ppm and above. Pei and Zhang [20] studied the adsorption of the toluene at 20%, 50% and 80% relative humidity. Results showed that the adsorption capacity of the activated carbon diminished upon increasing relative humidity, although the increase in concentration resulted in an increase in adsorption capacity. These results agreed with the results of our experiments. The saturation ratio curves for 20 ppm, 40 ppm and 80 ppm of toluene at T = 23 °C, $\phi = 50\%$ are shown in Fig. 11.



Fig. 11. Saturation ratio curves for 20 ppm, 40 ppm and 80 ppm of toluene at $T = 23 \text{ °C}, \phi = 50\%$

The adsorption of toluene on activated carbon took less time at 80 ppm than at 40 ppm and 20 ppm. The maximum saturation ratio and the time needed to reach was 90% at 190 min for 20 ppm, 95% at 105 min for 40 ppm and 95% at 75 min for 80 ppm. The saturation ratio curves for 20 ppm, 40 ppm and 80 ppm of toluene at T = 23 °C, $\phi = 70\%$ are shown in Fig. 12.



Fig. 12. Saturation ratio curves for 20 ppm, 40 ppm and 80 ppm of toluene at T = 23 °C, $\phi = 70\%$

The maximum saturation ratio and the time needed to reach it for the 20 ppm, 40 ppm and 80 ppm of toluene has an approximate value of 93% and 195 min, 95% and 110 min and 95% and 85 min, respectively. The saturation ratio curves for 20 ppm, 40 ppm and 80 ppm of toluene at T = 23 °C, $\phi = 90\%$ are shown in Fig. 13.



Fig. 13. Saturation ratio curves for 20 ppm, 40 ppm and 80 ppm of toluene at T = 23 °C, $\phi = 90\%$

During the first loading sequence, the maximum saturation ratio was evaluated at approximately 95% after 200 min for 20 ppm, 92% after 125 min for 40 ppm and 100%

after 90 min for 80 ppm. The experiment was carried out with an initial toluene concentration of 40 ppm in raw air and a constant temperature of 23 °C, while the humidity varied from 50%, 70% and 90%.

As results from data presented in Figs. 8–11, higher humidity resulted in a longer time needed to reach to the maximum saturation ratio. At a humidity of 90% and 80 ppm of toluene, the system reached the maximum saturation ratio after 90 min, whereas under similar conditions but with a relative humidity of 50%, the system reached the maximum saturation ratio in a shorter time (75 min). Hu et al. [21] performed many experiments at 25 °C, 44% relative humidity and 10 ppm, 30 ppm and 50 ppm of toluene . Similarly, they found that upon increasing concentration, the time to reach the maximum saturation capacity of toluene at 10 ppm, 31 ppm and 70 ppm, and results showed that the time to reach the maximum saturation ratio diminished upon increasing concentration, which is similar with the results of our examinations. The saturation ratio curves for 20 ppm, 40 ppm and 80 ppm of toluene at T = 33 °C, $\phi = 50\%$ are shown in Fig. 14.



Fig. 14. Saturation ratio curves for 20 ppm, 40 ppm and 80 ppm of toluene at T = 33 °C, $\phi = 50\%$

The time to reach the maximum saturation ratio for 80 ppm of toluene is considerably lower than for 20 ppm and 40 ppm. The saturation ratio curves of different toluene concentrations show similar increasing trends up to the maximum saturation ratio. The maximum saturation ratio of toluene is found as 92% at185 min for 20 ppm, 100% at 100 min for 40 ppm and 100% at 65 min for 80 ppm. The saturation ratio curves for 20 ppm, 40 ppm and 80 ppm of toluene at T = 33 °C, $\phi = 70\%$ are shown in Fig. 15.

There is a rapid increase in the saturation ratio up to 190 min for the 20 ppm concentration, up to 105 min for 40 ppm and up to 75 min for 80 ppm, and then the saturation ratio tended to a constant value. After the system reached the maximum saturation point (at 190 min) for 20 ppm, the saturation ratio became constant during the experiment. The saturation ratio curves for 20 ppm, 40 ppm and 80 ppm of toluene at T = 33 °C, $\phi = 90\%$ are shown in Fig. 16.



Fig. 15. Saturation ratio curves for 20 ppm, 40 ppm and 80 ppm of toluene at $T = 33 \text{ °C}, \phi = 70\%$



Fig. 16. Saturation ratio curves for 20 ppm, 40 ppm and 80 ppm of toluene at T = 33 °C, $\phi = 90\%$

As seen in Fig. 16, the maximum saturation ratio and the time needed to arrive to the maximum saturation point is found as 90% at 195 min for 20 ppm, 90% at 120 min for 40 ppm and 92% at 85 min for 80 ppm.

After the toluene–air mixture passes through the active carbon filter, the toluene concentration in treated air reaches high levels and continues in this range after the system arrived at the maximum saturation ratio for all test runs. In this case, at of 33 °C, 23 °C, 15 °C and toluene concentrations of 20 ppm, 40 ppm and 80 ppm, relative humidity has a remarkable influence on the amount of toluene adsorbed. A decrease is observed in the adsorption rate under increasing humidity, and therefore the time to arrive at the maximum saturation point became longer. Cal et al. [22] observed a reducing effect in the adsorption rate of acetone and benzene at a relative humidity of 50% and above. Pei and Zhang [20] and Owen et al. [23] identified negligible changes in the saturation ratio curve for a relative humidity up to 50%, a limited change for 75% relative humidity and significant change for 80% relative humidity.

In spite of that finding, at all concentrations and humidity ratio levels, increasing temperature increased the adsorption rate and shortened the time needed to reach the maximum saturation ratio probably due to the increase in the pore diffusion coefficient and resulting increase of the pore diffusion rate [14].

In the present paper, for a temperature up to 33 °C and 80 ppm of toluene, the time to reach the maximum saturation ratio decreased with an increase in temperature. However, Das et al. [24] examined saturation ratio curves in terms of gradually increased temperatures (35–100 °C) and high concentration rates (2000–10 000 ppm). The results of the study showed that below 50 °C, the time needed to arrive at the maximum saturation ratio increased upon increasing temperatures. However, a remarkable decrease in the time needed to arrive at the maximum saturation ratio was observed at 75 °C, and there is limited adsorption at higher temperatures above 100 °C. As understood from this, an increase in temperature shortens the time needed to achieve the maximum saturation ratio for low concentrations of toluene (4–80 ppm), whereas an increase in temperature up to 50 °C increases the time needed to arrive at the maximum saturation ratio for low concentrations (2000–10 000 ppm).

4. CONCLUSIONS

Coconut-based active carbon used in the present study is harder than wood-based activated carbon and has abundant functional groups on the surface [25]. It also has a finer pore size. The pore radius of the coconut shell-based activated carbon is less than 1 nm. Moreover, it has slit-like macropore structure showing better adsorption characteristics.

The adsorption rate of a substance is most effective when the pore sizes of the adsorbent and the size of the adsorbate molecules are similar. The adsorbate molecules are unable to enter too small adsorbate pores, too large pores partially capture too small molecules. Therefore, the efficiency of adsorption on activated carbon depends on the accessibility of its surface.

Experiments in the present study were performed for three temperatures at constant relative humidity and concentration. An increase in the temperature resulted in a decrease in the time needed to reach the maximum saturation ratio. The adsorption rate of toluene increased upon increasing temperature due to the increasing pore diffusion coefficient and resulting increased pore diffusion rate.

The time required to arrive at the maximum saturation point in dependence on humidity showed two different trends, depending on the concentrations. Increasing relative humidity resulted in a decrease in the time needed to reach the maximum saturation ratio for the low concentration levels. This occurred due to the duration of the experiment and low concentration of the toluene which allowed the pores to fill with moisture particles. However, increasing relative humidity increased the time needed to arrive at the maximum saturation ratio for high concentration levels. The total experiment duration was shorter at higher toluene concentrations. Yhe moisture particles enhanced the adhesion of toluene molecules onto the activated carbon macropores, as well as to the solid surfaces of the activated carbon.

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