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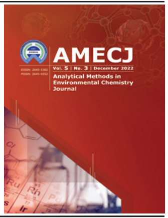

A rapid removal of xylene from air based on nano-activated carbon in the dynamic and static systems and compared to commercial activated carbon before determination by gas chromatography

Mostafa Jafarizaveh[a], Akram Tabrizi[a] and Farideh Golbabaei [b,*]

[a] *MSc. of Occupational Health Engineering, lecturer, Health Faculty, Gonabad University of Medical Sciences, Gonabad, Iran*
[b,*] *Professor of Occupational Health Engineering, Department of Occupational Health, Faculty of Health, Tehran University of Medical Sciences, Tehran, Iran*

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ABSTRACT

As main air pollutants, volatile organic compounds (VOCs) must be paid special attention. In this study, the removal efficiency of xylene from the air was investigated by nano-activated carbons (NACs) as an efficient adsorbent and compared to commercial activated carbons (ACs). In the chamber, the xylene vapor in pure air was generated, stored in the airbag (5 Li), and moved to adsorbents. Then, the xylene vapor was absorbed on the NAC/AC adsorbents and desorbed from it by a heat accessory. The efficiency of xylene removal with NACs and ACs was investigated in the dynamic and static systems based on 100-700 mg L^{-1} of xylene, flow rates of 100 ml $min^{-}1$, and 100 mg of adsorbent at a humidity of 32% (25°C). Xylene concentrations were determined by gas chromatography equipped with a flame ionization detector (GC-FID). In the batch system, the maximum absorption capacity for NACs and ACs was obtained at 205.2 mg g^{-1} and 116.8 mg g^{-1}, respectively. The mean adsorption efficiency for NACs and ACs adsorbents was obtained at 98.5% and 76.55%, respectively. The RSD% for NACs ranged between 1.1-2.5% in optimized conditions. The characterizations of the NACs adsorbent showed that the particle-size range was between 35-100 nm. The results showed that the adsorption efficiency of NACs for removing xylene from the air was achieved more than ACs. The GC-MS validated the proposed procedure in real air samples.

1. Introduction

Xylene is a colorless, aromatic hydrocarbon with a sweet taste that is easily flammable. Xylene is naturally present in petroleum and coal, and as a result of forest fires, it's produced some of it. Xylene is a toxic one-ring aromatic compound that its release in the air can have a significant effect on the health and well-being of humans and the quality of the air. The permissible exposure limit (PEL or OSHA PEL) for xylene is 100 ppm in the air [1]. One of the main concerns of industrial health experts is the control of volatile organic compounds such as xylene. Activated carbons are broadly defined to include the range of amorphous carbon-based materials and have a high degree of porosity with an extended surface area[2]. Recently, the removal of organic

*Corresponding Author: Farideh Golbabaei
Email: fgolbabaei@sina.tums.ac.ir
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compounds from the air by various processes such as adsorption, biofiltration and oxidation, has been widely studied. Gholamreza Mousavi et al showed that the xylene and other contaminated air such as benzene were removed from the air by a catalytic ozonation process. This study aims to evaluate the efficiency of activated carbon based on ozone (catalytic ozonation) to remove different concentrations of xylene. The results indicated that the efficiency of catalytic ozonation for removing xylene in the air was higher than single adsorption by AC [3]. In another study, the effect of retention time, ozone dose and relative humidity on the efficiency of the catalytic ozonation process in the removal of xylene from contaminated air were studied by Gholamreza Mousavi et al. They showed that the adsorption capacity of the AC adsorbent improved with the increase of inlet ozone dose as well as gas flow rate[3]. So far, a wide range of adsorbents has been used to separate various compounds from the air. These adsorbents differ in physical and chemical characteristics, such as the shape and size of the cavity, surface area, cavity volume, and surface activity. The superiority of nanoadsorbents over previous adsorbents is due to the nanometric scale, which causes tremendous changes in their physical and chemical properties. Other factors affecting the adsorption of analytes (BTEX) are the much number of adsorption sites and the extreme facilitation of molecular interactions in nanoadsorbents. Bin Gao et al reported developments of VOCs adsorption onto a variety of engineered carbonaceous adsorbents, including activated carbon, biochar, activated carbon fiber, carbon nanotube (CNTs), graphene (NG/NGO) and its derivatives (IL-NG), carbon-silica composites (CSC), ordered mesoporous carbon(OMC), etc. The key factors that influence VOC adsorption are analyzed with a focus on the physiochemical characteristics of adsorbents, properties of adsorbates, and the adsorption conditions[4]. Hamidreza Pourzamani et al investigated the adsorption capacity of nanoadsorbents to remove benzene and xylene from aqueous solutions. In this study, single-walled carbon nanotubes (SWCNTs), multi-walled carbon nanotubes (MWCNTs), and hybrid carbon nanotubes (HCNTs) were used. This study showed that the efficiency of nanoadsorbents was significant in removing xylene from the air [5]. In another study, the removal of Ortho, Meta, and para-xylene from the air samples was carried out on an oxidized carbon nanotube cartridge as an adsorbent by Le Huu Quynh Anh et al. In this study, the oxidized carbon nanotube with carbonyl groups significantly increased the adsorption capacity of xylene isomers[6]. In another study by Lu et al, the multi-walled carbon nanotubes were oxidized with sodium hypochlorite and used to remove ethyl benzene and para-xylene from aqueous solutions and significantly improved the removal of benzene and para-xylene[7]. Also, Golbabaei et al showed that nano-activated carbon adsorbent has a higher adsorption capacity for xylene removal compared to commercial activated carbon in the static state[8]. The adsorption of BTEX on carbon nanotube cartridges from air samples was reported by Le et al and the results showed that the CNTs had high potential for BTEX adsorption due to their microporous structure and high surface area[9]. They showed that the oxidized CNTs with carbonyl groups increased its adsorption capacity for these isomers [6]. Also, Shahi Ahangar showed that the photocatalytic removal efficiency in the concentrations of 50, 100, and 300 ppm was equal to 87.8%, 98.9%, and 90.8%, respectively [10]. Many researchers used different pilot and nanoadsorbents (Ni-MWCNTs, GQDs, NGO, NG, MSN, ILs, Silica) to remove hazardous pollutants such as toluene, ethyl benzene, mercury, benzene, dust, and hydrogen sulfide from the air [4,11-20]. Finally, according to those mentioned above and the high efficiency of nanoadsorbents compared to other adsorbents in the removal of different materials in the previous studies, a comparison study for adsorption of xylene from the air between commercial activated carbon absorbent and Nano carbon adsorbent was obtained to survey the effect of nanoadsorbent in the removal of xylene from the air. The previous studies for removing BTEX from the air reported a low efficiency in the air ambient.

In this study, NACs were used as adsorbent to remove xylene vapor from the air and compared to ACs (NIOSH adsorbent). All effective parameters were optimized. The xylene in pure air was generated in the chamber of pilot for the dynamic process. Also, the effect of temperature on the desorption/desorption of xylene and reusability of NACs or ACs adsorbents was studied. The retention time of nano-activated carbon was also studied and its efficiency was calculated over different days (inter-day and intra-day).

2. Experimental

In this experimental-analytical study, the feasibility of replacing activated carbon with nano-activated carbon for removing xylene vapors from the air was studied. The study was done according to the following steps.

2.1. Instrumental and Reagents

In this study, Gas chromatography equipped with a flame ionization detector was used (Varian 3800, CP7996 with a length of 25 m and a diameter of 0.25 mm). The FID detector was chosen for xylene analysis in gas/liquid. Before injection, the slide the plunger carrier down until it is completely over the syringe plunger, and tighten the plunger thumb screw finger-tight. The injector temperature was adjusted to 200°C. The column temperature reached from 35°C to 90°C with the speed of 20°C per minute, and the split ratio was adjusted from 2 to 1. The detector temperature tuned at 220°C. Hydrogen gas as a carrier gas was used at a flow rate of 30 ml min^{-1}. A Hamilton syringe was used for sample injection into the injector. Minimum and maximum pressures in psi for inlets and detectors tuned at the bulkhead fitting at the back of the gas chromatograph (100 psi). Finally, according to the peak areas of injecting of different xylene concentration to the GC-FID, a calibration curve was drawn (Fig.1). Agilent 5977B single quadrupole GC/MS based on cost-effective solution including the High-Efficiency Source (HES) for the most challenging detection limit samples was used for the xylene determination and the validation of procedure. Chemicals were acquired from Sigma Aldrich Germany. The mixed xylene (Catalogue Number**:** 108633, CAS N:1330-20-7, ACS, 99.5% purity) and para-xylene (Catalogue Number: 108684, CAS N:106-42-3, ACS, 99.7%) was procured from Merck. The standard solutions of xylene were made based on the procedure.In this study, the activated carbon (ACs) and Nano-activated carbon adsorbents (NACs) were prepared from Iranian Research Institute of the Petroleum Industry (RIPI, size <100 nm).

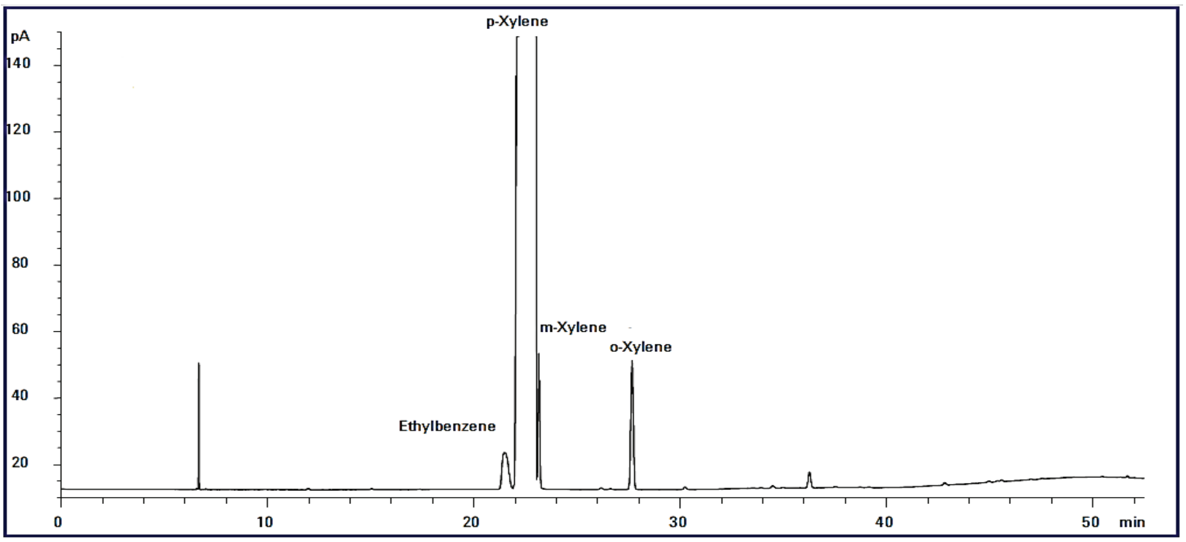

Fig.1. Peak area of GC-FID for species of xylene

2.2. Characterizations

The nano-activated carbon specification was investigated by Transmission Electron Microscopy (TEM), Electron Microscopy Scanning (SEM), and X-Ray Diffraction (XRD) at the Iranian Research Institute of the Petroleum Industry (RIPI). X-ray diffractions (PW 1840, Phillips, Netherland) based on radiation source of Cu-K_{α}. The morphologies of the NACs were achieved by scanning electron microscopy (SEM) and transmission electron microscopy(TEM), respectively (PW3710, CM30, Philips, Netherland). SEM and TEM of NACs showed Nano size below 100 nm.

2.3. Absorption procedure

In this study, according to the NIOSH method [21], various concentrations of xylene (purchased from Merck Company) were prepared in a range of 10 to 300 mg L^{-1} in sampling bags (Tedlar). Then, using a syringe, 400 microliters from the air existent in the sample bags were taken. In dynamic procedure,the different xylene concentration in pure air was made in the chamber of pilot. (Fig.2). The standard solution of xylene vapored in the chamber, mixed with pure air, and moved to a PVC storage bag (1-5 Li) at 25°C. GC-FID has measured the certified reference gas of xylene. Then the xylene gas moved to NACs sorbent and AC with a flow rate from 100 mL up to 300 mL per min. After xylene absorption on the NACs/ ACs adsorbents, the xylene was released from the adsorbent by heating up to 150°C. Then xylene concentration was determined by GC-FID. In the static system, the absorption xylene based on AC and NACs was achieved by a closed GC vial. In the static state, various concentrations of xylene were injected into the vial containing 5.0 mg adsorbent. Then 400 µl of air from the vial was injected into the GC, and the adsorption capacity of the NACs/ACs adsorbents was calculated according to Equation 1.

$$q_e = \frac{(C_0 - C_e) \times V}{m}$$

(Eq. 1)

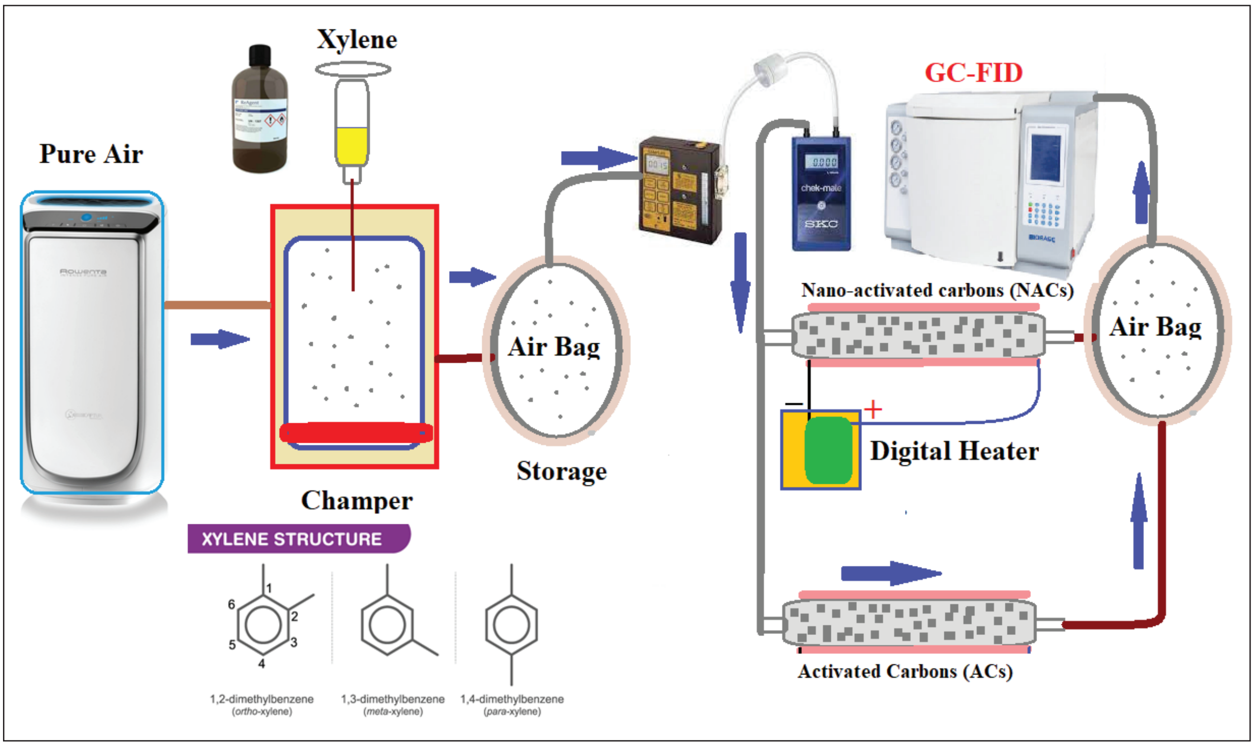

Fig.2. A schematic of the dynamic absorption system

3. Results and Discussion

3.1. Absorption efficiency and the effect of various factors on NACs

In this study, the different concentrations of 50, 200, 450, and 700 mg L-1 of xylene were injected by syringe into the chamber (Impinger) in presence of pure air. Then, the micro personal sampling pump (SKC, 20-300 ml min^{-1}) in different flows (100, 200, and 300 ml min^{-1}) passed the air containing xylene from the impinger into a sample bag (1 or 3 liters). In this experiment, at the beginning of every test, the impinger was heated to warm the air inside, and xylene was steamed completely. Finally, after filling the sample bag, the air inside it was taken by GC gas syringe and injected into GC-FID, and then, the results were analyzed. After assuring the mixing of xylene with inlet air into the designed pilot, the adsorbent was placed after the sampling bag. The amount of weight of adsorbent was 100, 150, and 200 mg. During this experiment, the adsorption of various concentrations of xylene in different air flows and amounts of adsorbent mass were investigated. The experiments were carried out at constant moisture and an ambient temperature (25 °C). It should be noted that these experiments were carried out for two activated carbon and Nano activated carbon, and adsorption efficiency was calculated after a sampling time of 30 minutes (Equation 2). Each experiment was repeated five times, and the efficiency was obtained below Equation 2.

$$\text{Efficiency (\%)} = (C_i - C_f) / (C_i) \times 100 \quad \text{(Eq. 2)}$$

C_i: initial xylene concentration and C_f: xylene concentration after passing from the adsorbent.

3.2. The storage stability of NACs

In this stage, to determine xylene adsorbed Nano-activated carbon, the 300 mg L^{-1} of xylene was passed on absorbent (100 mg) with a flow of 100 ml min^{-1}. After the adsorption of xylene on NACs or ACs, two ends of the absorbent tube were sealed with paraffin and stored at 0 °C, and the absorbent was analyzed at different times (in terms of days), and the efficiency was calculated according to Equation 3

$$\text{Removal Efficiency (RE)} = A_f / A_i \times 100 \quad \text{(Eq. 3)}$$

(A_f= Final analysis for xylene concentration adsorbed on adsorbent after distinct duration A_i**:** initial analysis for xylene concentration adsorbed on adsorbent).

3.3. Repeatability of NACs

Nano-activated carbon (NACs) recovery and its reusability were studied in this study. For this purpose, the nano activated carbon adsorbent was used in the dynamic system. The adsorbent's efficiency was obtained in a concentration of 300 mg L^{-1} from xylene in a flow rate of 100 ml min^{-1} and 100 mg of adsorbent. After xylene adsorption on NACs adsorbent, the xylene desorbed from NACs by the thermal accessory. The results showed that the recovery decreased after 16 times of adsorption/desorption. So, 16 times was considered as the number of reusability of adsorbent. The analysis for NACs or ACs was done three times (Mean of three determinations of samples ± confidence interval; P = 0.95, n =10). In this study, the mean values of the results are presented.

3.4. Data analysis and images

To analyze the data and compare the efficiency of various adsorbents for the removal of xylene, the SPSS software version 16 was used. Nano-activated carbon was analyzed utilizing TEM, XRD and SEM. The XRD, SEM, and TEM of activated carbon (ACs) and Nano activated carbon (NACs) are shown in Fig. 3a-e. The particle size of Nano-activated carbon was obtained below 100 nm by SEM and 30 nm by TEM. Also, XRD images showed a cubic structure of Nano activated carbon and activated carbon.

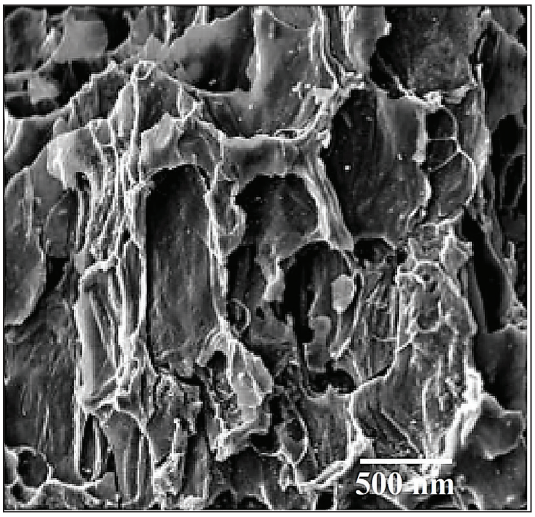

Fig.3a. SEM of activated carbon (ACs)

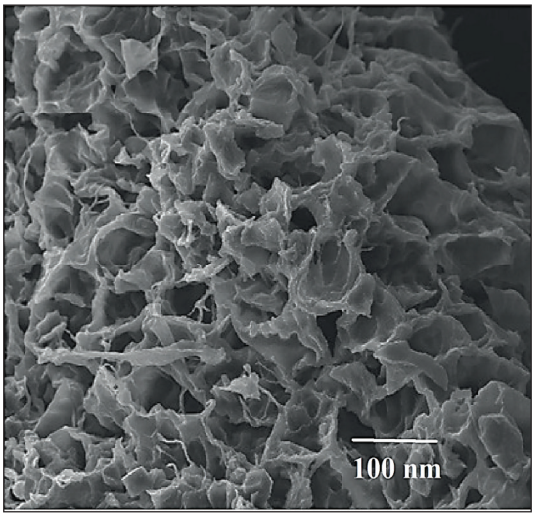

Fig.3b. SEM of Nanoactivated carbon (NACs)

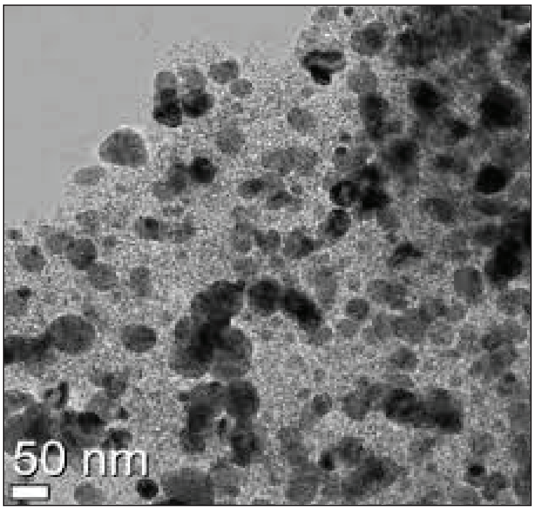

Fig.3c. TEM of activated carbon (ACs)

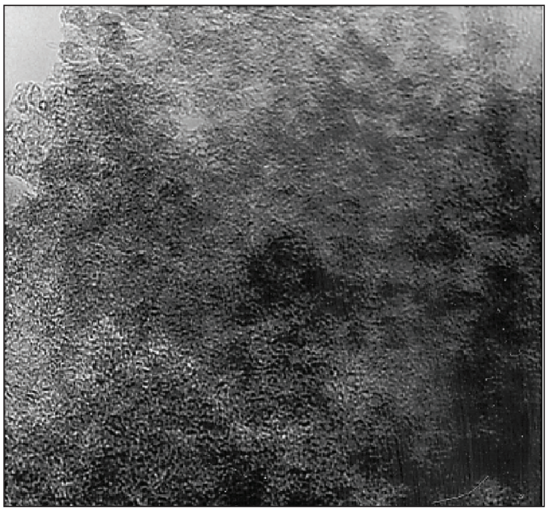

Fig.3d. TEM of Nanoactivated carbon (NACs)

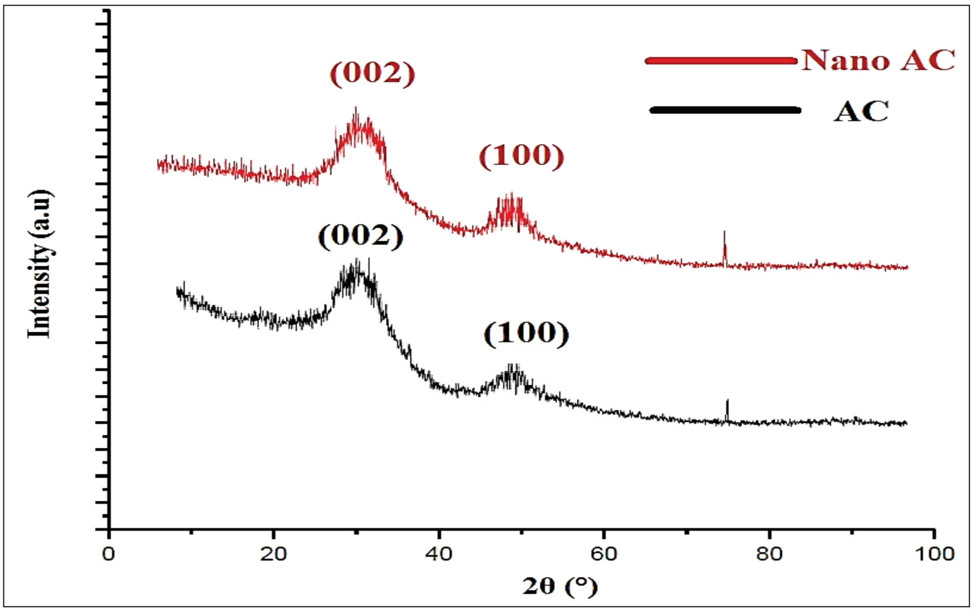

Fig.3e. XRD of activated carbon (ACs) and Nano activated carbon (NACs)

3.5. Adsorption Performance

The descriptive analysis of the adsorption efficiency (%) for NACs and AC is shown in Table 1. As can be seen, the average value of the adsorption efficiency of NACs is higher than AC (respectively 98.48 and 76.55). The statistical analysis of the results showed that the difference between the mean efficiency in Nano activated carbon and activated carbon was insignificant. (P-value =0.474). Linear regression was used to analyze the variables. In this analysis, the linear relationship between the dependent variable (adsorption efficiency, %) and independent variables (x1: xylene concentration, mg L^{-1}), (X2: air flow rate, ml min^{-1}) and (x3: adsorbent mass, mg) was investigated. The multi-linear regression equation was obtained as Equation 4 (R_{AC}: Activated carbon efficiency, R_{NAC}: Nano carbon Active Absorbance Adsorption Efficiency).

$$R_{CA}=75.598-0.027x_1-0.042x_2+0.087x_3$$

$$R_{NCA}=98.737-0.024x_1-0.057x_2+0.09x_3$$

(Eq. 4)

Regarding the nano activated carbon adsorbent, the value of R^2 was 0.9895 in the activated carbon adsorbent equal to 0.9524. The correlation between the adsorption efficiency of NAC and ACs obtained experimentally. The linear regression equation obtained from the adsorptions results of xylene by nano activated carbon and activated carbon adsorbents which were shown that there was a high correlation between them (correlation coefficient: 0.833) .

3.6. Effect of xylene concentration

In this study, the adsorption efficiency of xylene by NAC and ACs adsorbents in various xylene concentrations at optimized conditions such as the flow rate of 100 ml min^{-1} and 200 mg of absorbance was evaluated. First, the adsorption efficiency was constant and then, decreased by increasing the xylene concentration. The adsorption efficiency in the activated carbon adsorbent (ACs) was decreased significantly more than NACs adsorbent. At concentrations of 50, 100, 450 and 700 mg L^{-1}, the removal efficiency of NACs and ACs for 10 analyses was obtained (99.8%, 99.4%, 97.6% and 95.8%) and (77.4%, 75.9%, 69.2% and 60.1%), respectively.

3.7. Effect of flow rate

According to the results, the adsorption efficiency was decreased with an increasing flow rate. The effect of flow rate on activated carbon efficiency is higher than Nano activated carbon. At flow rates of 100, 200, and 300 ml min^{-1} with 200 mg adsorbents and xylene concentration of 100-200 mg L^{-1}, the removal efficiency of nano-activated carbon was equal to 98.5%, 94.3%, and 90.6%, respectively and for ACs was achieved 76.5%, 67.3% and 52.6%, respectively (Fig.4).

Table 1. Descriptive and analytical analysis of adsorbent efficiency of Nano activated carbon and activated carbon (P-value=0.474)

Adsorbent type	Number	AEA	SD	SE	CI(95%)	
					Lower limit	Upper limit
NACs	36	98.48	10.27	1.7	97.2	99.8
AC	36	76.55	10.03	1.67	67.4	85.7
Average	72	87.49	10.08	1.18	82.3	92.75

AEA: Adsorption Efficiency Average
SD: Standard Deviation
SE: Standard Error
CI: Confidence Interval

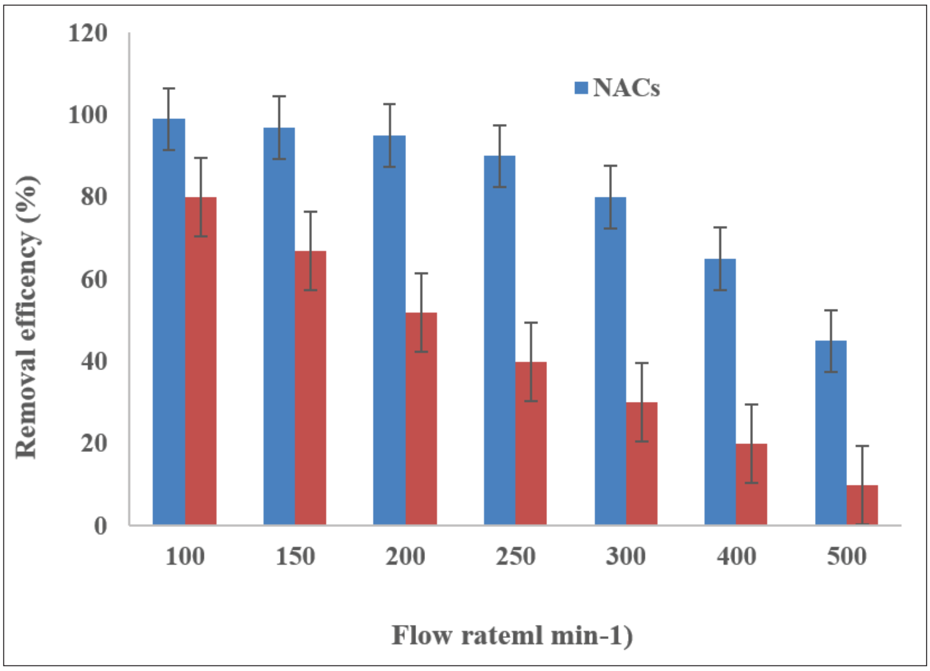

Fig.4. Effect of flowrate on removal efficiency of xylene by NACs and ACs adsorbents

3.8. Effect of adsorbent mass

According to the result, by increasing adsorbent, the removal efficiency of xylene from air increased and then constant. At the adsorbent mass of 100, 150 and 200 mg and concentration of 200 mg L^{-1} and flow rate of 100 ml min^{-1}, the efficiency of Nano activated carbon was 96%, 97.5%, and 98.5%, respectively. So, 100 mg of NAC was used as optimum amount of adsorbent. (Fig.5)

3.9. The effect of flow rate based on the amount of adsorbent

Figure 6 shows the combined effect of flow rate and the amount of adsorbent mass on the adsorption efficiency of nano activated carbon at various concentrations. As shown in Figures 5 and 6, by increasing amount of adsorbent and decreasing airflow rate, the adsorption efficiencies were increased.

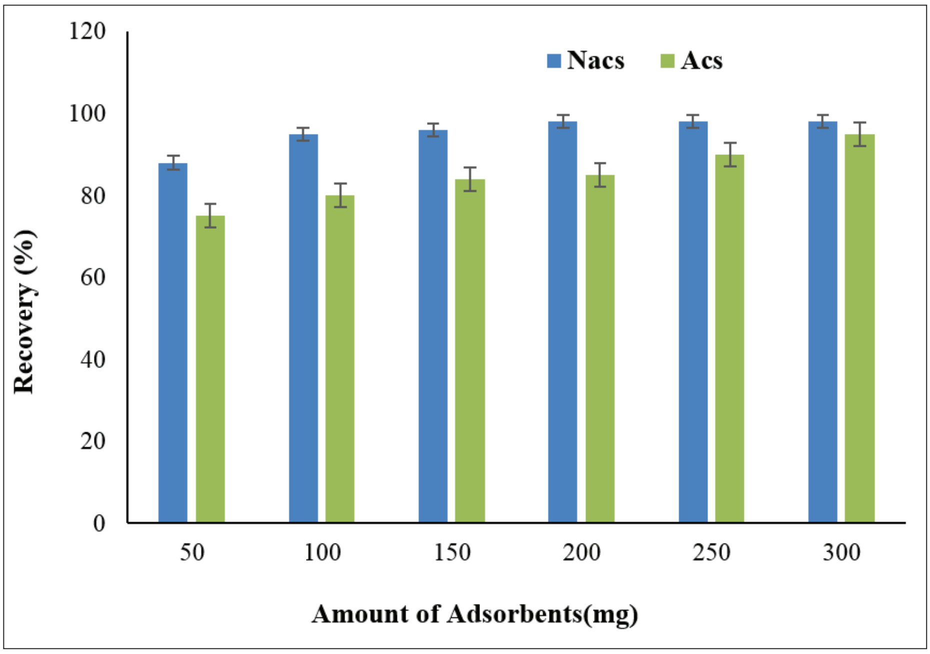

Fig.5. Effect of amount of NACs and ACs adsorbents on removal efficiency of xylene

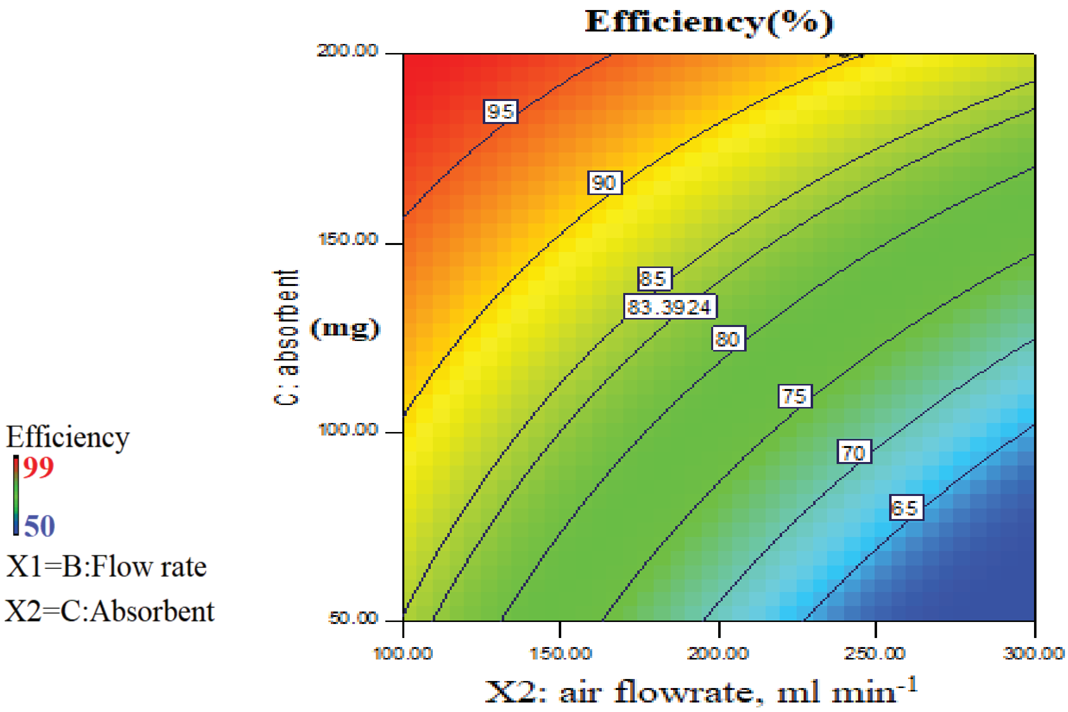

Fig. 6. Combined Effect of airflow and adsorbent mass on the adsorption efficiency of NACs

3.10. The effect of retention time

The effect of retention time of NAC on the xylene removal from air was investigated. Table 2 shows the xylene values added to the NACs and ACs adsorbents after passing pure air containing xylene vapor over it at a flow rate of 100 ml min^{-1} on different days (1, 3, 7, and 21 days). Then, the removal efficiency of the NACs and ACs adsorbents was calculated. The results showed that the retention time on nano-activated carbon was significant.

3.11. Effect of reusing and storage of adsorbent

By thermal accessory, the adsorbent recovered for 30 minutes. After 16 times of adsorption/desertion, the adsorbent efficiency was from 95.9%-99.3% for primary absorbent. After storing the closed original adsorbent in the refrigerator, it reached 94.5-96.1%. So, it is desirable with good recovery for removal of xylene by NACs in optimized conditions for 10 days storage in the refrigerator (- 4^{0}C). After 10 days, the recovery decreased.

Table 2. Results of the retention time for xylene removal from air by NACs (P-value: 0.462-0.513)

Row	XM	AM	XM_2	FR	RT	XM_3	(%) Efficiency
1	8.6	100	108.2	100	1	108.15	99.3
2	8.6	100	108.12	100	3	108.06	99.2
3	8.6	100	108.3	100	7	108.19	98.6
4	8.6	100	107.95	100	21	107.82	98.3

XM_2: xylene value (mg L^{-1}) + adsorbent mass (mg)
XM_3: The xylene value(mg L^{-1}) + adsorbent mass after retention time (days)
FR: Flow rate (ml min^{-1})
RT: Retention time (days)
AM: adsorbent mass (mg)
XM: Xylene value (mg L^{-1})

3.12. Discussion

Activated carbon is one of the essential adsorbents for removing volatile organic matter. In this study, nano-carbon adsorbent's efficiency for removing xylene from air compared to activated carbon and other carbon structures. Due to results and comparison of efficiency between NAC and AC adsorbents, NAC adsorbent had better performance and capacity than activated carbon for removing xylene from the air in the optimized conditions (xylene concentration, flowrate, and amount of adsorbent). The average adsorption efficiency of the NAC adsorbent was higher than activated carbon adsorbent in the same conditions (NAC:98.5% and AC:76.5%). The results showed that the difference in efficiency was not statistically significant (p-Value = 0.474). Due to previous studies, the efficiency of nano absorbents such as CNTs, GO, NG, and CQDs was higher than activated carbon. Golbabaei et al showed that the adsorption capacity of nano-activated carbon for xylene is higher than the activated carbon in the static state. There is a significant difference between the adsorption capacity and recovery of the two adsorbents. In another study by Golbabei et al, the results showed that the Nanographene had a higher adsorption capacity than nano graphene oxide and activated carbon adsorbents for removing xylene from the air. Also, they showed that the adsorption efficiency of nano adsorbents such as NAC, NG, NGO, and CNTs was higher than AC for removing xylene from the air [22]. Tanju Karanfil et al reported the adsorption of volatile organic pollutants by Nanographene sheet, which was compared to the carbon nanotube, activated carbon, and Nanographene. They introduced these alternative adsorbents to remove industrial organic compounds from water. This study also shows the high performance of the Nano adsorbent [23]. The results showed that increasing the air flow rate and the xylene concentration decreased the removal efficiency. On the other hand, the removal efficiency increased by adding the amount of adsorbents such as NAC or AC. This is due to an increase in retention time and available adsorption sites. Also, the results showed that a flow rate had a more significant effect on nano-activated carbon than activated carbon. This effect of the airflow rate caused no significant difference between the adsorption efficiency of the activated carbon and the nano-activated carbon. In fact, the study shows that low flow rates give better adsorption because the increase in flow rate actually creates a turbulent stream and the xylene molecules do not have enough time to adsorb onto the adsorbent. The results of flow rate and amount of adsorbent in the present study were similar to the results of Asilian et al. Asilian et al showed that the absorption capacity of xylene for zeolite was decreased up to 1.69 mg g^{-1} by increasing flow rate. Also, in a high flow rate, the time of failure and saturation time decreased in zeolite adsorbent [24]. Asilian also reported that increasing the amount of adsorbent cause to increase the adsorption efficiency. In fact, in this study, adsorption efficiency in low flowrate is was excellent and consistent [24]. Lu et al showed that the carbon nanotube oxidized with sodium hypochlorite had a higher BTEX adsorption than carbon nanotube and granular activated carbon. They showed that the physical and chemical properties of carbon nanotubes such as purity, structural and surface nature after oxidation were significantly improved, which led to a significant increase in BTEX adsorption capacity. Also, Shirkhanloo et al reported that the adsorption capacity of ionic liquid modified on nanographene caused to increase in the toluene removal from the air. The absorption capacity of toluene for IL-NGO was obtained at 126 mg g^{-1} which shows the remarkable favorite efficacy of nanoadsorbent for removing of volatile organic compounds. The modification of nanoadsorbent increased the potential of toluene removal [25]. In the present study, according to the results of experiments in different conditions, the nano-activated carbon showed a higher removal efficiency than activated carbon and competed with the IL/NGO or IL/CNTs. One of the critical factors for absorbents is the ability to recycle them. Nano adsorbents are the most economical and recyclable. By the proposed procedure, the results showed that the nano-activated carbon can also be recovered by heating and can be reused many times.

4. Conclusion

During the review of previous studies, it has been found that this study is the only one conducted to evaluate the efficiency of nano activated carbon adsorbent in removing xylene. This study showed that the nano activated carbon adsorption efficiency (98.5%) was somewhat higher than the activated carbon (76.5%), this difference was statistically significant. The absorption capacities (AC) for the NACs and ACs adsorbents ranged from 187.6-245.2 mg g^{-1} and 89.7-132.4 mg g-1, respectively (n=10, m=100 mg). According to the results, both absorbents have the potential to be used for removing xylene from the air with favorite efficiency and capacity. As a suggestion, the NACs as a novel adsorbent can be used optimally and more studies are needed in this regard.

5. Acknowledgment

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