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Vaseline-loaded expanded graphite as a new adsorbent for toluene

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ABSTRACT

A new kind of vaseline-loaded expanded graphite (VEG) complex material was prepared by a thermal soaking method and characterized by Brunauer–Emmett–Teller surface area, contact angle meter, scanning electron microscopy and mercury porosimeter. The use of VEG as an adsorbent for the control of toluene vapors in air was investigated at different humidities and compared with commercial granular activated carbon (GAC). The results showed that the VEG was much more hydrophobic than the original expanded graphite and could adsorb more toluene. The amounts of loaded vaseline on the expanded graphite surface were optimized and the VEG-2 was the best one. For example, the sorption capacity of 1 g of the VEG-2 was 213 mg of toluene, more than 4.5 times that of the expanded graphite. The high adsorption capacity of the VEG could be attributed to the partitioning action of vaseline for toluene. It was also found that the VEG for toluene was independent on the humidity of the streams, while the sorption capacity of the GAC was strongly dependant on humidity. Therefore, it was expected to have a longer durability than GAC in wet weather.

1. Introduction

Volatile organic compounds (VOCs) include aliphatic, aromatic and chlorinated hydrocarbons etc., which are emitted into the atmosphere mainly from human activities, such as production of adhesives, paints, printing materials and chemicals for synthesis [1]. VOCs directly or indirectly contribute to environmental issues, such as photochemical reactions creating urban smog, and cause heath problems. For all these reasons, a considerable effort has been dedicated in the last years regarding the removal of VOCs.

Many approaches have been used to eliminate VOCs, including plasma [2], catalytic oxidation [3], biological treatment [4,5] and adsorption [6]. Among them, adsorption is an attractive technique because of its simplicity in configuration and convenience in operation. Activated carbon (AC) is a most widely used adsorbent for VOCs because AC has various developed pores and large surface areas etc [7–9]. However, there are rich oxygen-containing groups, such as C=O, –OH, etc., on the surface of AC [10–16]. The water vapor in streams is easily adsorbed on these polarized functional groups. The adsorbed water will greatly decrease the sorption capacity of AC for hydrophobic VOCs [17,18]. The competitive adsorption between water and VOCs becomes a serious problem for AC used to remove hydrophobic VOCs [19]. Thus, it is necessary to develop a new kind of adsorbent with the high hydrophobic property and excellent adsorption capacity for hydrophobic VOCs.

Expanded graphite (EG) is a well-known material usually produced from various graphite intercalated compounds. By far, EG has been used in many fields, such as electromagnetic interference shielding, thermal insulation, electrochemical applications, stress sensing and adsorbent for heavy oil [20]. Among these applications, the potential as an adsorbent for heavy oil has attracted much attention due to the surprisingly high sorption capacity of EG for various oils. Studies showed that the maximum adsorption capacity of 1 g of EG reached as high as 86 g of A-grade heavy oil or 76 g of crude oil [21,22]. It was reported that the pores of EG are mainly macropores [21,23]. Lillo-Ródenas et al. found that toluene was mainly adsorbed into micropores, except for some mesopores [24], therefore, VOCs, like toluene, cannot be effectively adsorbed by EG. However, EG can adsorb oil and oil can be able to absorb hydrophobic VOCs, such as toluene, according to the principle of similarity and intermiscibility. Then it is reasonable to assume that EG firstly adsorbs oil and the oil-loaded EG can eliminate hydrophobic VOCs. This idea aroused our curiosity to develop a new adsorbent based on EG to remove hydrophobic VOCs.

During preliminary research, vaseline was chosen as one of oils due to its low cost as a by-product of oil refining. The aim of this paper was to approach the loading method of oils on EG and the adsorption performance of the new adsorbent for VOCs. The roles of vaseline and EG in the new adsorbent were also studied, especially vaseline. Considering that toxic toluene is an easily volatile solvent

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Table 1
Properties of the GAC-1 and the GAC-2.

Sample	BET surface areas ($m^2\,g^{-1}$)	Pore diameter (nm)	Total pore volume ($cm^3\,g^{-1}$)
GAC-1	893	2.3	0.55
GAC-2	856	3.4	0.48

Table 2
Amounts of polarized groups on the surface of the GAC-1 and the GAC-2.

Sample	Carboxylic groups ($mmol\,g^{-1}$)	Phenols ($mmol\,g^{-1}$)	Lactonic groups ($mmol\,g^{-1}$)
GAC-1	0.18	0.12	0.08
GAC-2	0.32	0.26	0.11

used frequently in many industries, it was chosen as a model of VOCs in the present paper.

2. Experimental

2.1. Materials

Vaseline was obtained from Tianjin Chemical Reagent Co. Ltd. Graphite intercalated compounds were purchased from Hebei Laiyin Company. Volume meter was obtained from Zhejiang Yuyao Meter Company. Toluene and nitric acid with analytical-grade were supplied by Guangzhou Chemical Reagent Company. Granular activated carbon (GAC) was obtained from Tianjin Fuchen Reagent Co. Ltd. with 80 mesh of the GAC particle.

2.2. Preparation of samples

EG was prepared from graphite intercalated compounds by a reported process in the literature [25]. Then EG was added into the mixture of vaseline and benzene under stirring at 60 °C for 5 min, and the ratio of EG (g), vaseline (g) and benzene (mL) was 1:10:5. The mixture (EG, vaseline and benzene) was separated by heat filtration, and then the mixture was placed at ambient temperature for 2 days. The different VEGs were obtained by changing the ratio of vaseline to EG.

The GAC was oxidized by HNO_3 solution ($HNO_3:H_2O = 1:5$, v/v) for 4 h at room temperature and washed by distilled water until the filtrate was neutral. The GAC-1 was an original sample and the GAC-2 was an oxidized one. The BET surface areas, the average pore sizes and the total pore volumes of the GAC-1 and the GAC-2 are presented in Table 1.

2.3. Characterization of samples

Specific surface area calculated using Brunauer–Emmett–Teller (BET) equation and Barrett–Joyner–Halenda (BJH) method for pore size distribution were determined at 77 K under a nitrogen atmosphere with a Micromeritics ASAP 2010 apparatus. Contact angles for 2 μL water droplet on the surface of EG and the VEG were measured with contact angle system OCA (Dataphysics Co., Germany) at ambient temperature in air. The scanning electron microscopy (SEM) was obtained on gold-coated samples using a JSM-6330F-mode Field Emission Scanning Electron Microscope (JEOL, Japan). The total pore volumes of EG and the VEG were measured by mercury porosimeter (Poremaster 60, USA).

The oxygenated surface groups on the GAC-1 and the GAC-2 were determined according to the method of Boehm in the literature [26]. The samples were dried at 110 °C overnight and cooled to ambient temperature in a desiccator before experiment. A known mass of 1.00 g of the GAC was added to 50.00 mL of one of three following bases of 0.05 M concentration: $NaHCO_3$, Na_2CO_3 and NaOH. The samples were agitated by shaking for 24 h and then filtered to remove the GAC, and 10.00 mL filtrate was taken by pipette from each sample and acidified by the addition of 30.00 mL of 0.05 M HCl, to ensure complete neutralization of the base. The acidified solutions were then back titrated with 0.05 M NaOH. Color indicators were used to show the endpoint [26]. Fresh solutions were used to limit the effect of CO_2 on the results. The amounts of oxygen-containing polarized groups on the GAC-1 and the GAC-2 are presented in Table 2.

2.4. Adsorption experiments

The dynamic adsorption test was carried out at 20 ± 0.5 °C. The flow chart of adsorption reaction is presented in Fig. 1. The toluene gas was blew from an closed flask containing liquid toluene by N_2 flow. The mixture of N_2 and toluene gas with a fixed humidity passed through a Pyrex pipe (length 150 mm, inner diameter 12 mm) filled with about 1.50 g of sample. The concentration of toluene was 70 $mg\,L^{-1}$ and analyzed by gas chromatography (GC, Shimadzu GC-2014). The volume flow rate through the adsorber was 25 $L\,h^{-1}$. The following equation can be used to calculate the amount of adsorbed toluene at the adsorption equilibrium:

$$w = \frac{F}{M}\left(c_0 t - \int_0^t c_i\,dt\right) \tag{1}$$

where w is the amount of adsorbed toluene; F is the volume flow rate; C_i and C_0 are the outlet and inlet concentration of toluene, respectively; M is the mass of adsorbent; and t is the adsorption time.

3. Results and discussion

3.1. Preparation and characterization of the VEG

The vaseline can be adsorbed into worm-like EG particles as heavy oil [21–23] and the different VEGs were prepared by using different ratio of vaseline to EG. Fig. 2 shows the dependence of vaseline content and the pore volume of the VEG on the ratio of

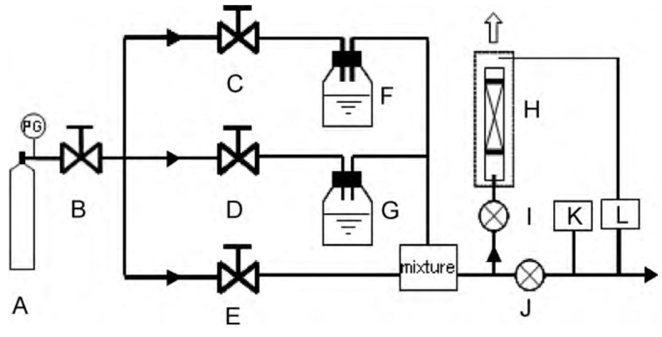

Fig. 1. Schematic setup of adsorption experiment: (A) N_2 gas; (B–E) valve; (F) water; (G) liquid toluene; (H) adsorber; (I) and (J) flow meter; (K) temperature and humidity meter; (L) GC; PG, pressure gauge.

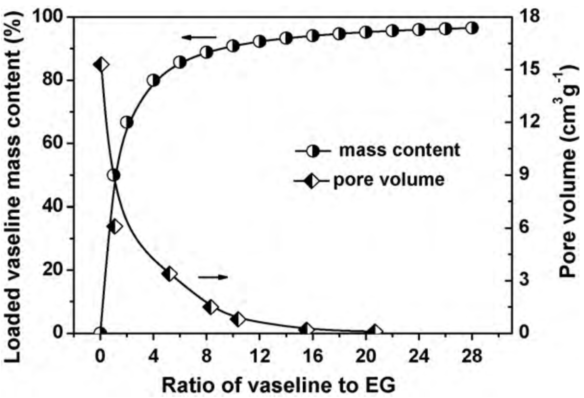

Fig. 2. Effects of the ratio of vaseline to EG on the vaseline content and the pore volume of the VEG.

Table 3
Specific surface areas (SSA) and contact angles of the different samples.

Sample	EG	VEG-1	VEG-2	VEG-3
Ratio of vaseline to EG	0	5	10	15
SSA ($m^2 g^{-1}$)	33.8	1.69	1.48	0.89
Contact angles (deg)	97 ± 1	110 ± 1	112 ± 2	120 ± 2

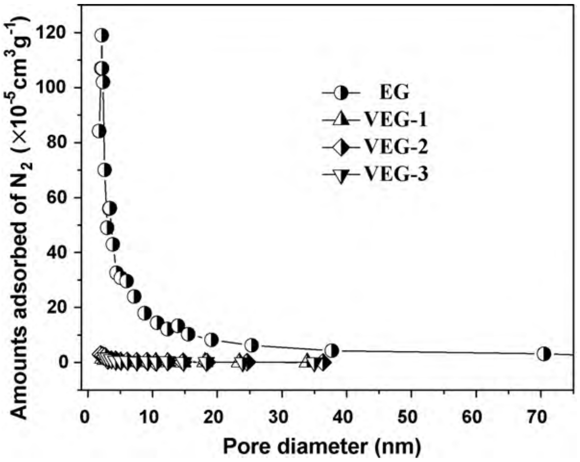

Fig. 4. Pore size distributions of EG with the different ratios of vaseline to EG.

vaseline to EG. It can be seen that the vaseline content rapidly increased with increase in the ratio and then slowly reached a platform as the ratio was beyond 20. This indicates the maximum sorption amounts of vaseline per 1 g of EG was 20 g under our experimental conditions.

The SEM images of EG and the VEG were presented in Fig. 3. The SEM image of EG shows that there were plentiful wedged pores on the surface of EG (Fig. 3A), which was in agreement with the results of the literature [25]. After modification, vaseline was adsorbed into the wedged pores, and there was a vaseline layer on the surface of EG (Fig. 3B). In the present experiment, the pore volumes of the different VEGs were measured by mercury porosimeter. The variation of the VEG volume with increasing the amount of the loaded vaseline was shown in Fig. 2. It is obvious that the volumes of the VEG were rapidly decreased with increase in the amount of the loaded vaseline.

On the contrast, the maximum vaseline-loaded amounts of 1 g of zeolite (∼3 mm) and 1 g of vermiculite (∼3 mm) were measured at the identical experiment conditions and were only 0.15 and 0.18 g, respectively. The total pore volumes of zeolite and vermiculite measured by mercury porosimeter were 0.1 and 0.2 $cm^3 g^{-1}$, respectively, far smaller than that of EG (15.3 $cm^3 g^{-1}$ in Fig. 2). Therefore, EG can be loaded a larger amount of vaseline attributing to its large pore volume measured by mercury porosimeter, which was in agreement with Toyoda's et al. work. In addition, it was found that the sorption amounts of oil were greatly dependent on the total volume of EG measured by mercury porosimeter [21].

Compared with EG, the specific surface areas of the VEG considerably decreased with increase in the vaseline-loaded amount (Table 3). The specific surface area of EG was 33.8 $m^2 g^{-1}$, which was in agreement with the reported results [27,28], while that of the VEG-1, the VEG-2 and the VEG-3 decreased to 1.69, 1.48 and 0.89 $m^2 g^{-1}$, respectively. It can be seen from Fig. 4 that the surface of the EG only possessed some pores in the range of 3–4 nm, while the VEG did not process pores in our experimental conditions. This might be ascribed to vaseline layer blocked the pores on the EG surface.

Fig. 5 shows the shapes of a dye-containing water droplet on the surface of EG and the VEG-3, respectively. It can be seen from the photographs that the contact angle of the VEG-3 was greater than that of EG. Moreover, the increase in the contact angle was related to the vaseline-loaded amount (Table 3). If the contact

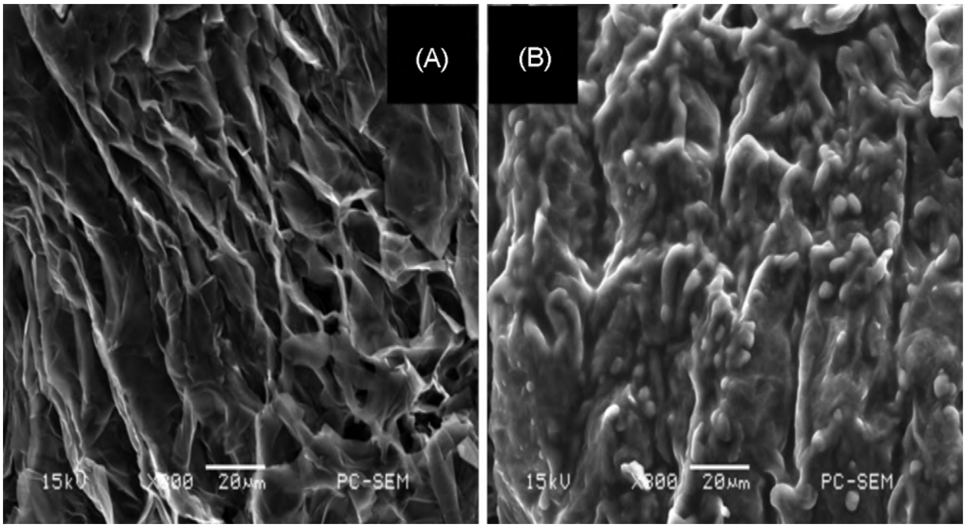

Fig. 3. SEM micrographs of EG (A) and VEG (B).

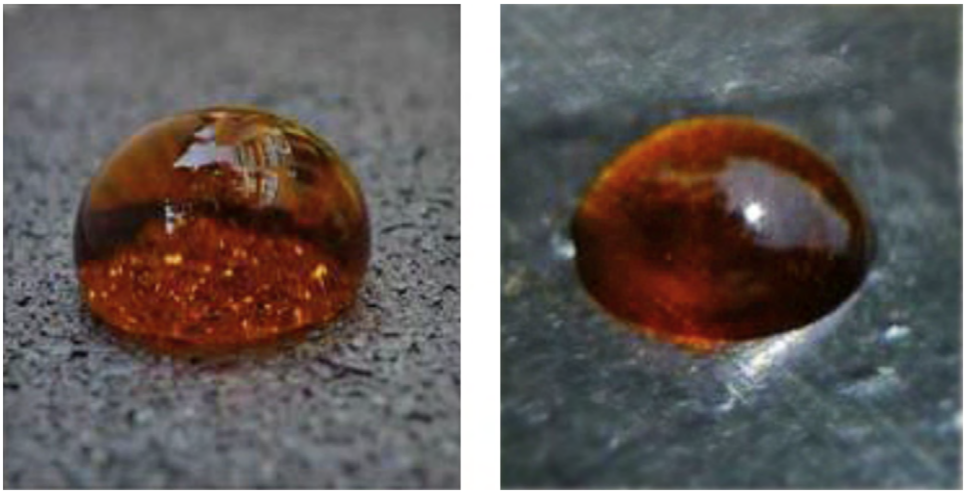

Fig. 5. Shape of a dye-containing water droplet on the surface of the VEG (left) and EG (right).

angle was regarded as an indicator of hydrophobicity, the data in Table 3 shows the surface hydrophobicity of the VEG-3 increased by 23.7% in comparison with that of EG. The increase in the surface hydrophobicity of the VEG leads to the increase in the adsorption capacity of the VEG for hydrophobic VOC [28] and decrease in the competitive effects of water in streams.

3.2. *Adsorption characteristics of VEG for toluene*

Fig. 6 presents the dependence of the amount of the adsorbed toluene on the vaseline mass content in the VEG. It shows that the sorption capacity of the VEG considerably increased with increase in the vaseline mass content. For example, 1 g of the VEG-2 (the vaseline content was 90.9%) could adsorb 213 mg of toluene, while 1 g of EG could adsorb 38.5 mg of toluene. The sorption capacity of the former was 5.5 times as much as that of the latter at the identical conditions.

The relationship between the amount of the adsorbed of toluene and the vaseline content in the VEG can be described by a linear equation (2), as shown in the insert of Fig. 6:

$$W = 196m + 40.5 \tag{2}$$

where W is the amount of the adsorbed of toluene by 1 g of the VEG, m is the vaseline mass content in the VEG. From the equation, it can be seen that the sorption capacity for toluene could increase by 19.6 mg as the vaseline content increased by 10% in the VEG, indicating the sorption capacity of the VEG was greatly dependent on vaseline content in the VEG.

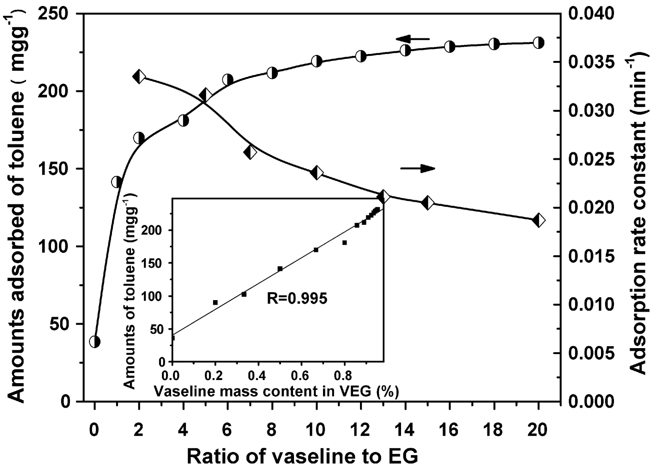

Fig. 6. Effects of the ratio of vaseline to EG on the amount of adsorbed toluene and the uptake rate constant of the VEG.

Generally, the adsorption capacity and the uptake rate constant of the adsorbents are very important parameters for the engineering design in actual application. From above discussion, we have known that the amount of loaded vaseline had a great effect on the sorption capacities of the VEG. Therefore the effect of the vaseline-loaded amount on the uptake rate constant of the VEG for toluene was also investigated. In our experiment, the adsorption rate of the VEG for toluene could be described by the linear driving force (LDF) kinetic equation (3) [16]:

$$\frac{Q_t}{Q_0} = 1 - e^{-kt} \tag{3}$$

where Q_t is the amount of the adsorbed toluene at time t, Q_0 is the maximum amount of the adsorbed toluene at the identical experimental conditions, k is the uptake rate constant, and t is the adsorption time.

It can be seen from Fig. 6 that the uptake rate constant of the VEG for toluene was intensively dependent on the vaseline-loaded amount. But with increase in the ratio of vaseline to EG, the adsorption amount of toluene was increased, while the uptake rate constants decreased. And when the ratio was below 10, the adsorption capacities of the VEG rapidly increased and then increased very slowly, while the uptake rate constant decreased smoothly with increase in the ratio of vaseline to EG. Considering both of the adsorption capacity and the uptake rate constant, the VEG-2 with the ratio of 10 would be a proper one in actual application.

As we know, the competitive adsorption between VOCs and water is an important problem in practice and it is well documented in the literatures [29–32]. In order to investigate the competitive adsorption performance of VEG between VOCs and water, here the breakthrough time of the VEG-2 for toluene were investigated at different relative humidity and compared with that of the GACs, as shown in Figs. 7 and 8.

The samples used in Fig. 7 were outgassed overnight at 110 °C before experiment and cooled to room temperature in a desiccator. The samples in Fig. 8 were firstly outgassed at 110 °C for 12 h and then pre-humidified overnight at the relative humidity of 60% or 90%. From Tables 1 and 2 it could be seen that after oxidation the BET surface area, the average pore size and the total pore volume of the GAC-1 changed a little. It was noteworthy that after oxidation the polarized groups on the GAC-2 surface increased markedly, which was in agreement with the results of the literatures [33,34].

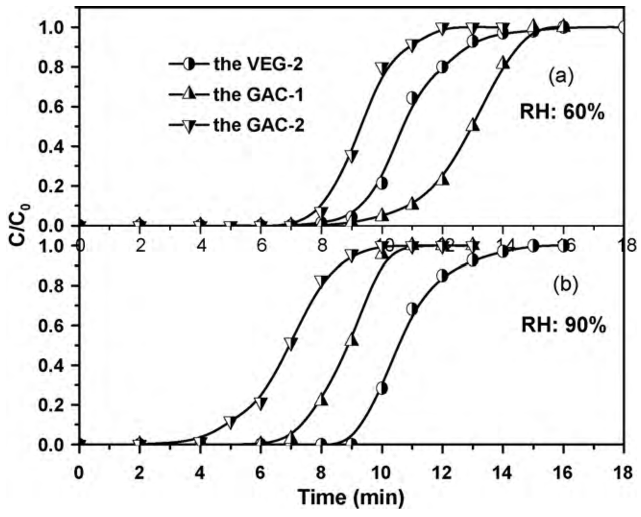

Fig. 7. Effects of relative humidity (RH) on the breakthrough time of the VEG-2 and the GACs.

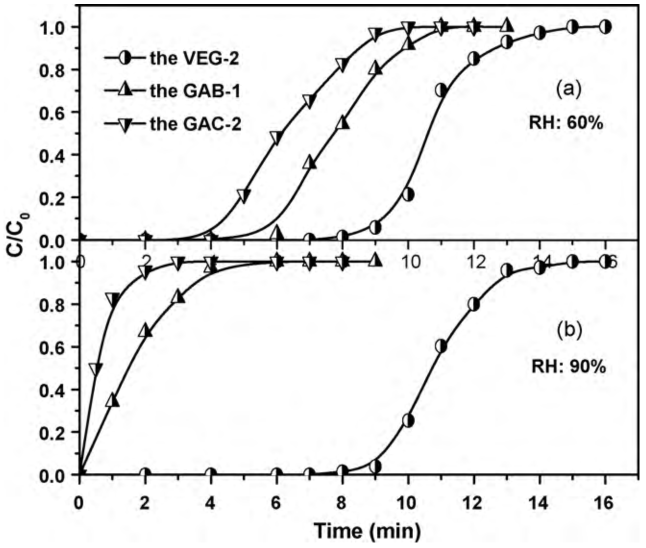

Fig. 8. Effects of relative humidity (RH) on the breakthrough time of the VEG-2 and the GACs.

The breakthrough times and the sorption capacities under different conditions are shown in Table 4. The breakthrough time in all of our experiments was the time when the outlet concentration of toluene in streams was equal to the incoming concentration. It could be seen that the breakthrough time and the sorption capacities of the VEG-2 did not change with the different relative humidity, while that of the GAC-1 and the GAC-2 decreased with increase in the relative humidity. In addition, it was noted that when the samples were pre-humidified, the breakthrough times and the sorption capacities of the VEG-2 were hardly unchanged whatever the relative humidity was, while that of the GACs decreased greatly as shown in Fig. 8.

Table 4
Breakthrough time and adsorption capacities of samples.

Sample	Results of Fig. 7				Results of Fig. 8			
	60%[a]		90%[a]		60%[a]		90%[a]	
	BT[b]	AA[c]	BT[b]	AA[c]	BT[b]	AA[c]	BT[b]	AA[c]
VEG-2	15	211	14	215	15	215	15	214
GAC-1	15	275	11	186	11	171	6	38.3
GAC-2	11	181	10	148	10	137	3	20.1

[a] The relative humidity of the streams.
[b] The breakthrough time of toluene in min.
[c] Amounts adsorbed of toluene by samples in mg g^{-1}.

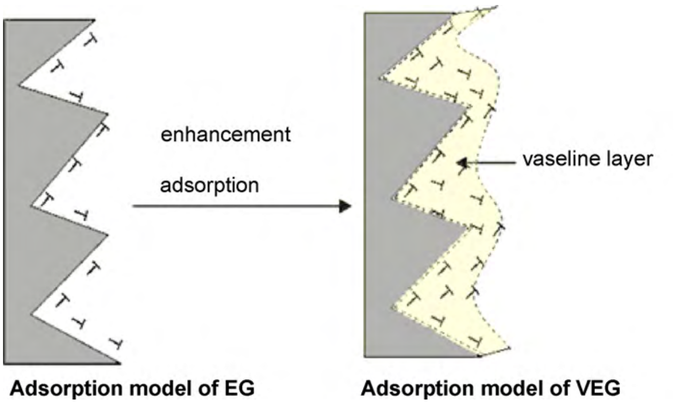

Fig. 9. Schematic description of proposed model for adsorption enhancement of toluene (T: toluene molecule).

The reason was contributed to the surface properties of the VEG and the GACs. The competitive adsorption of water and toluene took place synchronously [19,29]. And the negative effect of the water adsorbed via hydrogen bounding increased with increase in the relative humidity of the streams and the numbers of the polarized groups on AC surface [35,36]. From Tables 1 and 2 it could be seen that there were polarized groups on the surface of the GAC-1 and the GAC-2. But for the VEG it was totally different. From the SEM images of the VEG, it was found that there was a vaseline layer on the surface of the VEG and there was no polarized group on the surface of the VEG because vaseline was a hydrocarbon. Therefore water in streams could not be adsorbed on the surface of the VEG through hydrogen bounding and then the breakthrough time and the sorption capacities of the VEG-2 were not influenced by the humidity in streams.

From the view of the actual application, the regeneration and the reuse of toluene-saturated VEG are generally needed. In the paper, the method of heating regeneration was employed to regenerate the VEG. The used VEG-2 sample was heated at 80 °C about 3 h and the adsorbed toluene can be removed completely after heating. Our experiment results showed that the sorption capacity of the VEG-2 for toluene basically kept constant in the process of 20-run adsorption/desorption. This indicated that the VEG possessed a favorable regeneration characteristics and a good durability.

3.3. *Proposed adsorption mechanism*

Generally, besides adsorbates, the adsorption capacity of AC depends on its specific surface areas, the pore size distribution and the surface chemical properties [3,5]. It has been known from the above discussion that the VEG processed the small specific surface areas and nonporous structure and had an excellent adsorption capacity for toluene. This above contradiction suggested that VEG possessed a different adsorption mechanism from that of AC. According to the principle of similarity and intermiscibility, vaseline and toluene are all organic compounds, therefore, vaseline layer on the EG surface can partition toluene from flow and the toluene molecules was adsorbed into the vaseline layer of the VEG. Therefore the adsorption capacity of the VEG for toluene was enhanced. And the sorption capacity of the VEG was related to the amount of the loaded vaseline. The physical model of adsorption enhancement from EG to VEG was described in Fig. 9.

4. Conclusions

The VEG was first prepared by a thermal soaking method. It was found that the VEG was high hydrophobic and the VEG was nonporous and had small BET surface area. But the VEG processed an excellent sorption capacity for toluene. The adsorption capacity increased with increase in the vaseline-loaded amount, while the uptake rate constant decreased with increase in the vaseline-loaded amount. Considering both of them the VEG-2 with the ratio of 10 was a proper one in actual application. The sorption capacity of the VEG-2 was 213 $\text{mg}\,\text{g}^{-1}$, as high as 5.5 times that of EG at the identical conditions, and the uptake rate constant was 0.0238 min^{-1}. In addition, the sorption capacity of the VEG was almost independent on the relative humidity and kept invariable sorption capacity after 20-run heating regeneration. The enhanced mechanism of the VEG for toluene was mainly ascribed to the partitioning action of vaseline layer for toluene.

Acknowledgements

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