

A comparative investigation on absorption performances of three expanded graphite-based complex materials for toluene

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ARTICLE INFO

Article history:

Received 20 February 2010

Received in revised form 12 July 2010

Accepted 12 July 2010

Available online 21 July 2010

Keywords:

VOCs

Complex material

Unsaturated degree

Absorption

ABSTRACT

Three kinds of expanded graphite-based complex materials were prepared to absorb toluene by dispersing plant oil, animal oil and mineral oil on the surface of expanded graphite, respectively. These complex materials were characterized by scanning electronic micrograph, contact angle meter and Brunauer–Emmett–Teller surface area. And their absorption capacities for toluene were comparatively investigated. The results showed that the surfaces of the three types of sorbents were very hydrophobic and nonporous, but they all had excellent absorption capacities for toluene. And their absorption capacities were proportional to the toluene concentration in streams and decreased differently with increasing the absorption temperature. It was noteworthy that the absorption capacities varied with the unsaturated degree of the complex materials and kept unchanged under different relative humidities of streams. Moreover, the regeneration experiments showed that after 15-run regeneration the absorption capacities of expanded graphite modified by mineral oil almost kept unchanged, while that of expanded graphite loaded plant oil and animal oil dropped by 157 and 93.6 mg g⁻¹, respectively. The losses of their absorption capacities were ascribed to the destruction of their unsaturated carbon bounds.

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1. Introduction

Removal of volatile organic compounds (VOCs) has attracted much attention recently because VOCs have caused environmental and health problems, such as urban smog, destruction of ozone layer, infection to eyes, nose and even cancer [1–5]. Now many technologies have been developed to remove VOCs, including condensation, catalytic oxidations, biological treatments, adsorption and absorption. Among them, the absorption is a simple and effective technology [6–8].

Vegetal oil, diesel oil, silicone oil and solar oil have been used to absorb benzene, toluene, xylene, methanol, etc. [9–12]. But the absorption efficiency was greatly influenced by the contacting surface areas of streams to adsorbents and the viscosities of adsorbent. Therefore, to increase the contacting surface areas of adsorbent to streams would be one of the ways to efficiently improve the absorption efficiency of these oils [1,13]. Poddar et al. [10,14] proved that the coefficient values of the overall mass transfer of VOC were 3–11 times higher than those in conventional packed towers after increasing the contacting surface areas of streams to oil by membrane absorber.

Expanded graphite (EG) prepared from graphite intercalated compounds is a kind of hydrophobic carbon material and has varied macro- and mesopores [15–17]. As a new carbon material, EG has been used in many fields, such as electromagnetic interference shielding, thermal insulation, electrochemical applications, stress sensing and adsorbent for heavy oil [15]. EG has been proposed as an adsorbent for oil because of its surprising sorption capacity [18–20]. It was reported that 1 g of EG could adsorb 86 g of A-grade heavy oil or 76 g of crude oil [10,21]. And the work of Toyoda et al. showed that EG mainly processed macropores [19], therefore EG could not be directly employed as effective adsorbent for VOCs, like benzene or toluene, because these VOC molecules could be only firmly adsorbed into micropores and some of mesopores [21].

It is attractive that the incorporation of organic compounds into the framework of the porous materials will improve their adsorptive properties by increasing their affinity to organic compounds [22,23]. And the properties of the incorporated organic groups would influence the adsorption properties. Zhu et al. found that the adsorption amounts of organic compounds were related to the mass content of the organic groups [24]. Zhao et al. also found that the hydrophobicity and pore structures of the adsorbents had great effects on the affinity between adsorbents and adsorbates [25]. The work of Hu et al. showed that unsaturated degree of the organic group would influence adsorption capacity of adsorbents [22].

Considering that EG is a kind of excellent adsorbent for oil and some oils are identified to possess a potential absorption capacity for VOCs due to the principle of similarity and intermiscibility,

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consequently, it was expected that oil-loaded EG would be an interesting absorbent for hydrophobic VOCs. On the one hand, if oils are loaded on the surface of EG, the surface area of oil would be improved; on the other hand if the surface of EG was modified with oil it would be change to more hydrophobic than the virgin EG, which could decrease the negative effects of water vapor in streams on the sorption capacity of adsorbents for hydrophobic VOCs because the polarized functional groups on the surface of adsorbents, such as activated carbon, could easily adsorb water vapor from streams [26–29].

The paper was focused on comparative investigation on the absorption performance of various oil-loaded EG for VOCs under different conditions, such as temperature and relative humidity of streams. The aim of this article is mainly to find suitable oil from three kinds of frequently used oils (plant oil, animal oil and mineral oil) to prepare the complex absorbent. Considering that toluene is a kind of toxic solvent used frequently in many industries, it was chosen as a model of VOCs in the present paper.

2. Materials and methods

2.1. Materials

Graphite intercalated compounds was purchased from Hebei Laiyin Company, China. Mineral oil was obtained from Tianjin Chemical Reagent Co. Ltd., China. Animal oil and plant oil were obtained from market. Volume meter was bought from Zhejiang Yuyao Meter Company. Analytical grade toluene and benzene were supplied by Guangzhou Chemical Reagent Company, China. Iodine monochloride was purchased from sinopharm Chemical Reagent Co. Ltd., China. And the properties of mineral oil, animal oil and plant oil are presented in Table 1.

Plant oil, also known as triacylglycerol, comprises of 98% triacylglycerols and small amounts of mono- and diglycerides. Stearic, palmitic, oleic, linoleic and linolenic acids are fatty acids commonly found in plant oil [30]. The animal oil has high melting point and high viscosity, which results from higher stearic and palmitic acid contents [30]. Therefore there are lots of unsaturated carbon bounds in plant oil and animal oil. But mineral oil (namely vaseline) composes of saturated hydrocarbon.

2.2. Preparation of the complex materials

EG was prepared from graphite intercalated compounds using the procedure of the literature [31], EG was flexural and cylindrical powder with the length of ~ 8 mm and the diameter of ~0.2 mm. The ratio of EG (g), mineral oil (g) and benzene (mL) was 1:10:5. First vaseline and benzene melted and then was mixed with EG. After agitated for 5 min, the mixture of EG, mineral oil and benzene was placed at ambient temperature for 2 days and benzene would completely vaporize. At last MEG was prepared and only contained vaseline and EG. Plant oil or animal oil was used to prepare PEG or AEG with the identical process, respectively. Accordingly PEG only contained plant oil and EG and AEG only contained animal oil and EG. In the follow-

Table 1
Properties of selected oils.

Species of oil	Viscosity (Pa·s)	State (298 K)	Density (g·cm ⁻³)	Iodine value (g(100g) ⁻¹)
Plant oil	39.6 [30]	Liquid	0.903	102
Animal oil	^a	Semisolid	0.913	41.5
Mineral oil	^a	Semisolid	0.916	0.3

^a The animal oil and the mineral oil are semisolid at room temperature.

ing experiments the mass ratios of EG to oil was 1:10 if not specified.

2.3. Analysis and characterization

Specific surface area (SSA) of Brunauer–Emmett–Teller (BET) and pore size distribution of Barrett–Joyner–Halenda (BJH) method were determined at 77 K under nitrogen atmosphere with a Micromeritics ASAP 2010 apparatus (Micromeritics, USA). The morphology of complex materials was characterized by scanning electron microscopy (SEM, JSM-6330F-mode Field Emission). The samples were sprayed by gold. Contact angle for 2 μL water droplet on the surface of EG and modified EG were measured with contact angle meter at ambient temperature in air (Contact angle system OCA, Dataphysics Co., Germany). The concentrations of toluene in the inlet and outlet were measured by a GC (Shimadzu GC-2014) equipped with flame-ionization detector. The concentration of toluene was 80 mg L⁻¹ and the flow volume throughout the U-type Pyrex tube was 25 L h⁻¹. If not specified, the absorption temperature was kept at 293 ± 0.5 K in the experiments. The following equation could be used to calculate the amount absorbed of toluene at the absorption equilibrium:

$$w = \frac{F}{m} (c_0 t - \int_0^t c_i dt) \quad (1)$$

where w is the amount absorbed of toluene, F is the volume flow rate, c_i and c_0 are outlet and inlet concentration of toluene respectively, m is the mass of absorbent and t is the absorption time.

The method for determining the iodine values of samples used the national standard of China: animal and vegetable fats and oils-determination of iodine value (ISO 3961: 1996, MOD).

2.4. Experimental methods

The static absorption tests were carried out to investigate the absorption capacities of plant oil, animal oil, mineral oil, EG, PEG, AEG and MEG for toluene vapor. The experiment was carried out according to the reported method [32]. The experimental temperature was 293 ± 0.5 K and the concentration of toluene vapor was saturated at the experimental temperature.

The dynamic absorption tests were conducted to evaluate the continuous absorption performance of the complex materials and EG for toluene. The dynamic absorption set-up was presented in Fig. 1. The toluene vapor was blown with N₂ gas from a closed flask containing liquid toluene. The blown toluene vapor passed through an U-type Pyrex tube (length 230 mm, inner diameter 12 mm) filled with about 1.00 g complex material. And before absorption experiment the U-type Pyrex tube with absorbent was blown with N₂ gas for 2 h at 293 ± 0.5 K.

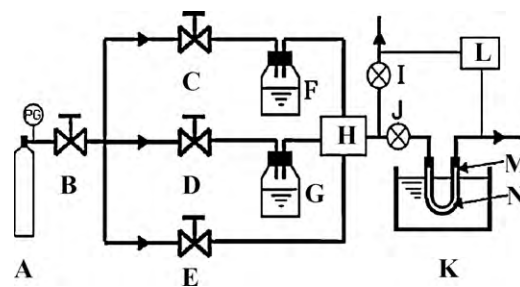


Fig. 1. Schematic diagram of experimental set-up. (A: N₂ gas; B–E: valve; F: liquid toluene; G: water; H: gas mixture; I and J: flow meter; K: temperature controller (ice/water bath) L: GC; M: U-type tube; N: absorbent; PG: pressure gauge).

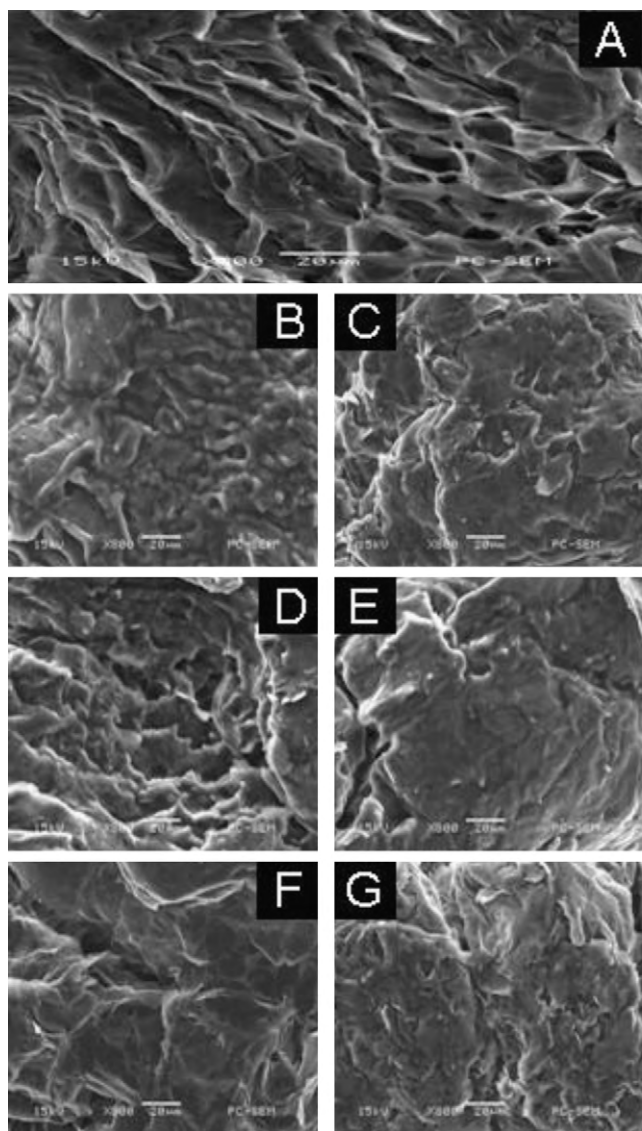


Fig. 2. SEM images of samples (A: EG; B: MEG; C: MEG after 15-run regeneration; D: AEG; E: AEG after 15-run regeneration; F: PEG; G: PEG after 15-run regeneration).

3. Results and discussion

3.1. Characteristics of complex materials

Three kinds of complex absorbents, MEG, AEG and PEG, were all prepared by the soaking method. Fig. 2 presents the SEM images of EG and these absorbents. It can be seen from these images that there were many wedged pores on the surface of EG (Fig. 2A), while these wedged pores filled by oil for MEG, AEG and PEG (Fig. 2B, D and F). After 15-run regeneration the SEM image of MEG, AEG and PEG were presented in Fig. 2C, E and G. From the SEM images it can be seen that there was still an oil film on the surfaces of three kinds of absorbents, respectively.

Table 2 shows the specific surface areas and contact angles of a water drop on the surface of EG, PEG, AEG and MEG, respec-

Table 2
SSAs and contact angles of the samples.

Sample	EG	PEG	AEG	MEG
SSA ($\text{m}^2 \text{g}^{-1}$)	33.8	1.78	1.35	1.69
Contact angle (degree)	97 ± 1	110 ± 2	111 ± 1	112 ± 2

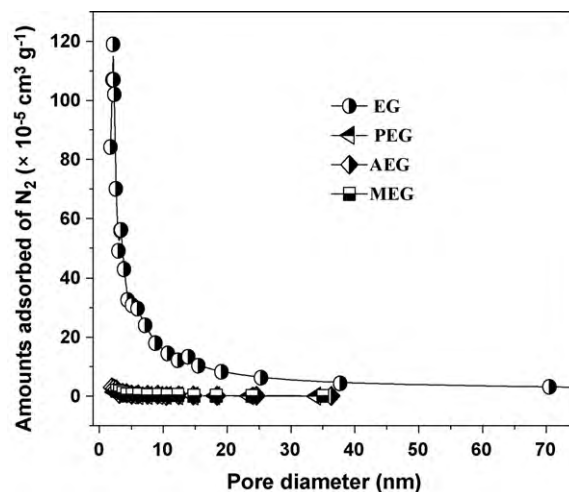


Fig. 3. Pore size distributions of EG, PEG, AEG and MEG.

tively. It is obvious that the specific surface areas of PEG, AEG and MEG decreased greatly, compared to that of EG, while their contact angles increased differently. The increase of the surface contact angle showed the surface of the new absorbents would be more hydrophobic than that of EG. In Fig. 3 the pore size distributions of EG, PEG, AEG and MEG show that EG possessed the mesopores with a pore size of 3–4 nm and there was no any mesopores on the surface of PEG, AEG and MEG. After modification oil filled the wedged pores and covered the surface of EG. Therefore the oil film blocked the mesopores on EG surface, resulting in the decrease of SSAs of PEG, AEG and MEG and the increase of their hydrophobicity because of the hydrophobicity of oil.

3.2. Static absorption characteristics of the complex materials

Table 3 shows the static absorption capacities of pure oils and oil-loaded EG (PEG, AEG and MEG) for toluene. The absorption capacities of 1 g of plant oil, animal oil and vaseline were 1053, 972 and 810 mg g^{-1} , respectively, while the oils were loaded on EG the absorption capacities of 1 g of pure plant oil, animal oil and vaseline were 1068, 966 and 817 mg g^{-1} under identical conditions, respectively. These data showed that the absorption capacity of oil for toluene was almost identical whether the oil was loaded on EG or not. This was because the absorption capacities of the complex materials were contributed to partitioning action of oil according to the principle of similarity and intermiscibility, while EG only played a role of supporter to disperse these oils.

Table 3
Sorption amounts of toluene under static condition at 293 K.

Sample	Sorption capacity (mg g^{-1})
MEG	817 ^a (743 ^b)
AEG	966 ^a (878 ^b)
PEG	1068 ^a (971 ^b)
Vaseline	810 ^c
Animal oil	972 ^c
Plant oil	1053 ^c
EG	85

^a The absorption capacities of 1 g of vaseline, animal oil or plant oil, which was loaded on EG, were calculated from the absorption capacities of 1 g of MEG, AEG or PEG divided by the mass content of oil in the complex materials.

^b The absorption capacities of 1 g of MEG, AEG or MEG.

^c The absorption capacities of 1 g of pure vaseline, pure animal oil or pure plant oil.

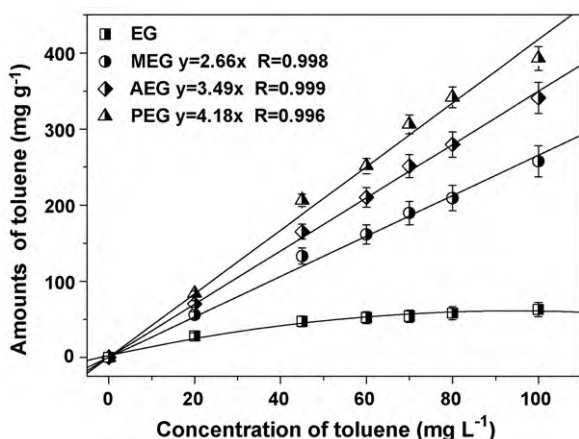


Fig. 4. Dependence of the amounts absorbed of toluene on the concentration of toluene.

3.3. Dynamic absorption characteristics of the complex materials

Fig. 4 shows the plots of the absorption capacities of EG, PEG, AEG and MEG for toluene against its concentration. It can be seen from the figure that the absorption capacities of PEG, AEG and MEG for toluene were linearly proportion to the concentration of toluene. The absorption capacity of PEG was always the largest one and the adsorption capacity of EG was always the smallest one at the identical concentration. Compared to that of the complex materials under static experiment, the absorption capacities of the complex materials under dynamic experiment were very small due to the difference of the toluene concentration and experimental conditions.

Iodine value is a good indicator to reflect the unsaturated degree of oil [33]. Experimental results showed that the iodine values of per 100 g of PEG, AEG and MEG were 101, 40.5 and 0.2 g, respectively. The unsaturated degree of complex materials were in the order of PEG > AEG > MEG, which agreed with the absorption amounts of toluene at the identical toluene concentration. Therefore the absorption amounts of toluene by three kinds of the complex materials seemed related to the unsaturated degree of oils.

Many works have showed that the non-specific interaction between adsorbents and adsorbates can increase the sorption capacity of adsorbents [34–36]. Vinh-Tham et al. found that the strong sorption of aromatic toluene by modified SBA-15 was caused by the strong interaction of π -electrons from the aromatic ring with the hydroxyl groups on the adsorbent surface [23]. The work of Hu et al. showed that the strong interaction between benzene and the phenyl groups on the surface of phenyl-SBA-15 increased its capacities for benzene through π -system interaction between aromatic rings of benzene and functional groups (methyl or phenyl groups) because the phenyl groups had stronger attractive interaction with benzene than the methyl groups [22]. In present experiment compared to the sorption capacity of EG, that of PEG and AEG improved and the reasons would be the stronger interaction via π -system interaction between aromatic rings of toluene and functional groups (unsaturated bounds). While the increase of the absorption capacity of MEG was ascribed to the interaction of π -system interaction between aromatic rings of toluene and alkyl groups in mineral oil. And the interaction between toluene and unsaturated bounds was stronger than that between toluene and alkyl groups [22]. Therefore PEG had the highest sorption capacity for toluene and MEG had the lowest one.

Fig. 5 presents the effects of absorption temperature on the sorption amounts of toluene. It could be seen that the absorption

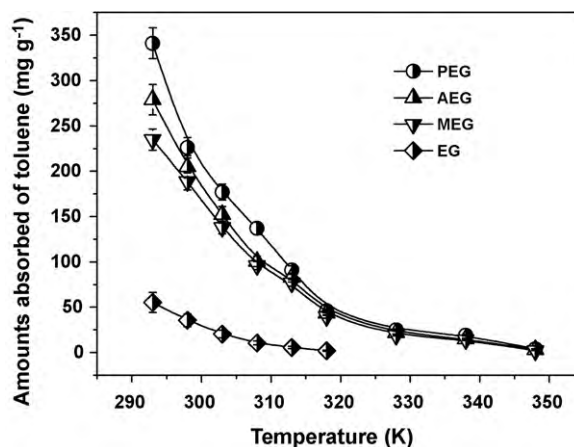


Fig. 5. Effects of temperature on the amounts absorbed of toluene.

amounts of toluene rapidly decreased with increasing the absorption temperature and then decreased slowly. This indicated that the three kinds of adsorbents were very sensitive to temperature. For example when the temperature was 293 K the uptake amounts of toluene by PEG, AEG and MEG were 341, 280 and 235 mg g^{-1} . When the temperature increased to 318 K, the absorption capacities of the complex materials for toluene dropped by 295, 236 and 195 mg g^{-1} , respectively. And from 318 to 348 K the absorption amounts of PEG, AEG and MEG dropped by 41.2, 39.3 and 37.3 mg g^{-1} , respectively. The decreasing degree of their sorption capacities was in the order of PEG > AEG > MEG, which agreed with their unsaturated degree. Therefore the results showed the sorption capacity of the prepared adsorbent with higher unsaturated degree dropped more than that of the prepared adsorbent with lower unsaturated degree.

According to the principle of similarity and intermiscibility, the oil film will absorb toluene by partition action. When the reaction reached equilibrium the relationship of the concentrations of toluene in the flow gas and oil film are related by Henry's law [9]:

$$H = \frac{C_g}{C_l} \quad (2)$$

where C_g and C_l are the equilibrium concentrations of toluene in the flow and modified EG, respectively and H is Henry's law constant.

When the temperature increases the coefficient of Henry's law will increase [37]. From Eq. (2) it can be concluded that when the concentration of toluene in flow, C_g is stable the concentration of toluene in modified EG, C_l will decrease. Therefore when the absorption temperature increased the absorption capacities of three kinds of complex materials decreased and from our experimental results it could be concluded that the more unsaturated degree the complex material was, the more the decrease of its sorption capacity.

Fig. 6 shows the effects of the relative humidity of streams on the absorption capacities of PEG, AEG and MEG for toluene. The results showed the relative humidity of streams had no influence on the absorption capacities of PEG, AEM and MEG for toluene. Generally, there were polarized groups on the surface of adsorbents, such as C=O and -OH [33,37]. All these polarized groups had an intensive affinity to water vapors in streams via hydrogen bonding. Therefore humidity of streams always influenced the sorption capacities of hydrophobic VOCs and the higher humidity would strengthen the negative effects of water [38,39]. But from above discussion the surface of the complex materials were hydrophobic and non-porous, water vapor in streams was hardly absorbed via hydrogen bonding.

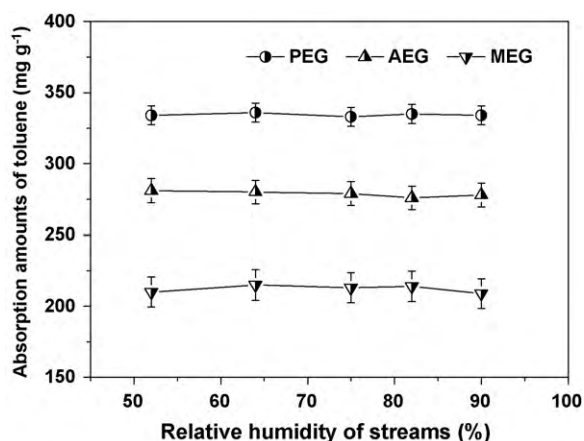


Fig. 6. Effects of relative humidity of streams on the amounts absorbed of toluene.

3.4. Repeated use

Fig. 7 presents the variation of the absorption amounts of toluene with regeneration run. The complex materials were regenerated through heating at 373 K for 2 h in constant temperature oven for 1-run. It was found that the absorption capacities of PEG and AEG for toluene decreased with increasing regeneration runs. After 6-run regeneration the absorption capacity of PEG for toluene was less than that of MEG, and after 10-run regeneration the absorption capacity of AEG was less than that of MEG. After 15 runs, the absorption capacities of PEG and AEG dropped by 157 and 93.6 mg g⁻¹, about 47.4% and 33.3% of the original absorption capacities of them, respectively, while that of the MEG almost kept unchanged.

The loss of the absorption capacities were mainly ascribed to the destruction of the unsaturated carbon bounds in oil molecules and the evaporation of oils on EG surface because of heating. Fig. 8 shows the variation of the iodine value with the regeneration runs. After 15-run regeneration the iodine values of 100 g of PEG and AEG dropped by 75.5 and 30.5 g, being 74.8% and 76.9% of the original iodine value of them, respectively, and that of MEG always kept about null because mineral oil used to prepare MEG was saturated. The mass losses of the three complex materials were only about 0.3%, 4.2% and 3.8%, respectively. So the decrease of the absorption capacities of PEG and AEG were greatly ascribed to the destruction of unsaturated bounds of oils. The unsaturated bounds were unstable and easy to be oxidized by oxygen during regeneration, leading to the decrease of absorption capacities of PEG and AEG. Fig. 9

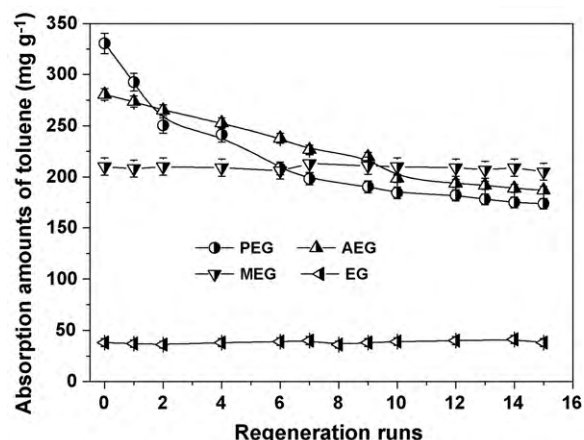


Fig. 7. Variation of the uptake amounts of toluene with the regeneration run.

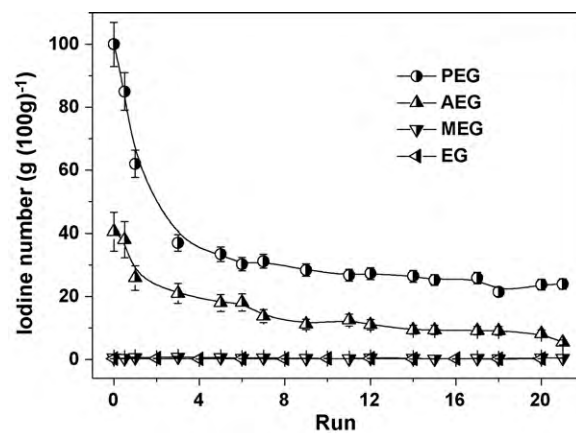


Fig. 8. Dependence of the iodine values of complex materials on regeneration times.

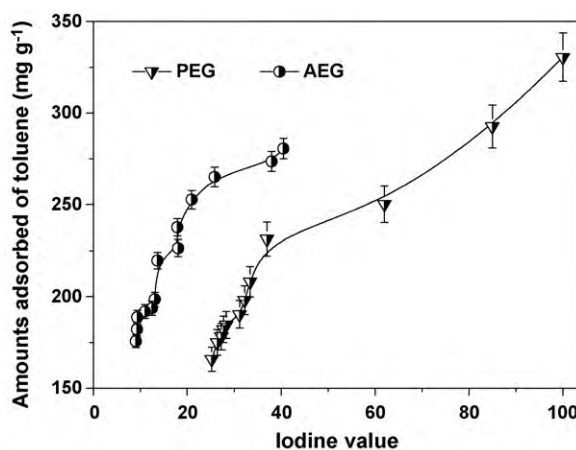


Fig. 9. Dependence of the absorption capacity on the iodine values of complex materials.

presents the variation of absorption capacities of PEG and AEG with their iodine values. It can be seen that the absorption capacities of PEG and AEG for toluene were greatly related to their iodine values. The higher iodine values of the adsorbent were, the higher its sorption capacities. The iodine value of MEG was null so during 15-run regeneration its absorption capacities almost kept unchanged because saturated mineral oil was difficult to be oxidized under experimental conditions.

4. Conclusions

PEG, AEG and MEG were prepared by dispersing oils on the surface of EG and their absorption capacities for toluene under different were comparatively investigated. The absorption capacities of PEG, AEG and MEG were linear with the concentration of toluene in streams. And the absorption capacities of PEG and AEG for toluene increased with increasing their unsaturated degree. When the absorption temperature increased the absorption capacities of PEG, AEG and MEG dropped quickly and the decrease rate of PEG was the largest one and that of MEG was the smallest one. It was noteworthy that the absorption capacities of three adsorbents kept unchanged under different relative humidities of streams. After 15-run regeneration the absorption capacities of PEG and AEG decreased by 157 and 93.6 mg g⁻¹, respectively, while that of the MEG almost kept unchanged because the mineral oil was saturated. The decrease of the absorption capacities of PEG and AEG were mainly ascribed to the oxidation of unsaturated bound in oils.

Acknowledgement

This research was supported by Foundation for University of Dongguan City 2010, Nature Science Foundations of China (50578163, 20877025); the Special Foundation of Sun Yat-Sen University; Science and Technology Research Programs of Guangdong Province (0711220600311) and Nature Science Key Foundations of Guangdong Province (92510027501000005).

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