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Adsorption of Polycyclic Aromatic Hydrocarbons onto Activated Carbon from Non-Aqueous Media: 1. The Influence of the Organic Solvent Polarity

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Abstract: Polycyclic aromatic hydrocarbons (PAHs) can be formed easily during the refinery processes of crude petroleum. Their accumulation poses serious operating problems and their removal is of great importance. In this investigation we tested the ability of activated carbon to remove a number of the PAHs compounds from mixtures of organics solvents, with different chemicals structures and polarities. Batch adsorption tests were used to investigate the effect of chemical structures and polarities of solvents on the adsorption of naphthalene, anthracene, and pyrene on activated carbon. Our investigation revealed that aromatic solvents have high affinity for activated carbon and therefore, inhibit PAHs adsorption, while polar solvents have low affinity for activated carbon and consequently the adsorbent sites are more available for the PAHs molecules. This behavior can be explained by the fact that the PAHs and benzene molecules are able to form π–π complex between π- electrons of benzene rings and active sites on an activated carbon surface. An

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increase in the molecular weight of aliphatic solvents such as hexane and heptane has little effect on adsorption of PAHs to activated carbon. However, cyclic hydrocarbon solvents such as cyclohexane increase the adsorption of small PAHs and decrease the adsorption of heavier PAHs molecules, probably as a result of differences in solubilities.

Keywords: PAHs adsorption, activated carbon, non-aqueous media, equilibrium isotherms

INTRODUCTION

PAHs commonly refer to a large class of organic compounds containing two or more fused aromatic rings made up of carbon and hydrogen atoms. PAHs are chemically inert, soluble in many organic solvents, and are highly lipophilic. Therefore, the behavior and the biological effects of PAHs vary in the environment (1). PAHs with low molecular weights (e.g., naphthalene and phenanthrene) are highly mobile in an aquatic environment and present significant acute toxicity to many organisms. Many PAHs of higher molecular weights (e.g., benzo(a)pyrene) have been shown to be highly carcinogenic and have significant persistence up to many years in the environment (2). On the other hand, some of these PAHs have a certain industrial importance (e.g., in dyes, plastics, pharmaceuticals, and the pesticides industry) (3).

PAHs are found naturally in crude oil, creosote, coal tar, and coal. The release of PAHs have been reported during production and processing, of plasticizers, dyes, and pigments; however, most PAHs enter the environment via the atmosphere from incomplete combustion processes, such as the processing of coal and crude oil during refining, coal gasification, and coking. Crude petroleum oil is also distinguished by the relatively high ratio of compounds with saturated five–membered rings and the presence of highly strained molecules, such as 4,5-methylene phenanthrene (4).

Many techniques (e.g., bioremediation (5–9), ozonation (10–15), photodegradation, and adsorption (16) have been applied successfully for the minimization of PAHs in wastewater of domestic and/or industrial plants and soils. The adsorption techniques are the most frequently used in batch mode for the minimization of such complex species in liquid or gas phases due to its high efficiency and applicability to be used at high temperatures. Activated carbon was considered the most frequent and effective solid adsorbent in this field. Other adsorbents (e.g., alumina, zeolite, fly ash, soils, and clays (17–20) have also been employed successfully to remove PAHs.

The formation of small amounts of PAHs from hydrocracking reactions can build up to concentrations that cause fouling of cooled heat exchanger surfaces, equipment, and fluid lines. The PAHs can build up to the limit of their solubility at temperatures commonly used for the condensation and the separation of the hydrocracker output products. The fouling problem may also gradually reduce the heat transfer to some extent that conversion and/or feed rate must be reduced. In the light of present trends towards heavier feedstocks, the adsorption, solubility, and precipitation of PAHs are consequently of great interest in the petroleum industry (21, 22). The removal of these compounds from exhausted gases has been studied by Mastral et al. (23–25). However, the studies on the adsorption of PAHs from non-aqueous media are scarce. Therefore, the objective of this work is to investigate the possibility of the minimization of PAHs compounds from organic media using activated carbon. Further, more different factors affecting the adsorption process of PAHs from non-aqueous media will be investigated.

EXPERIMENTAL

Reagents and Materials

A commercial activated carbon (CAC) manufactured by BDH, chemical laboratory supplies (Poole, BH15 1TD, England) was used as a solid adsorbent without further treatment and activated in a vacuum oven at 473.15 K overnight. The BET surface area, the average pore diameter, and other physical parameters of the CAC are summarized in Table 1.

High purity (98–99%) PAHs, namely naphthalene (NA), anthracene (AN), and pyrene (PY) were obtained from Merck. The solid adsorbates were dissolved in the required hydrocarbon solvents to simulate the media of hydrocracker output fluids. BDH (UK) and Fluka (Switzerland) organic solvents, namely hexane, n-heptane, cyclohexane, benzene, and methanol were used without further purification. Benzene was used as the base unit for the PAHs compounds. The non- polar solvents hexane, heptane, and cyclohexane represent the major components in the hydrocracker output fluids. Methanol was used to investigate the influence of solvent polarity on the adsorption of PAHs onto the CAC. The PAHs used in this study and their solubility in mole fraction are listed in Table 2.

Table 1. Physical properties of the CAC adsorbent

Property	Value
BET surface area, m^2/g	734
Total pore volume, cm^3/g	0.4335
Average pore diameter, ^oA	23.6
Bulk density, g/L	500
Particle size, μm	850–1700
Ash content, % (w/w)	5–8

Table 2. Mole faction solubility for the PAHs in different solvents at 298–299 K

PAHs/ solvent	Methanol	Hexane	Heptane	Cyclohexane	Benzene	Ref.
Naphthalene	0.0235	0.1168	0.1300	0.1487	0.2946	(27, 28)
Anthracene	0.00024	0.00129	0.00157	0.00157	0.00742	(29)
Pyrene	0.00149	0.00852	0.01101	0.01089	0.06316	(30, 31)

Apparatus

A thermostated water bath shaker (model SB-16, Techni Inc. UK) equipped with a temperature controller (Tempter Junior TE-8J) was used in the batch experiments. A Pye-Unicam double beam UV-Visible spectrophotometer (SP400, UK) was used for measuring the absorbance of each PAH at λ_{max} versus solvent blank. The adsorption of a pure solvent in its gaseous state onto a CAC adsorbent was critically investigated using the apparatus given in Fig. 1. The apparatus is composed of two reservoirs one for the adsorbent (1) and the other one for pure solvent (2). The two reservoirs are connected to each other via two valves (3 and 4) and a manifold was connected to a vacuum pump through a valve (5).

Recommended Batch Sorption Procedures

To investigate the effect of the solvent polarity on the adsorption of the different PAHs onto the CAC adsorbent, the adsorption isotherms of each

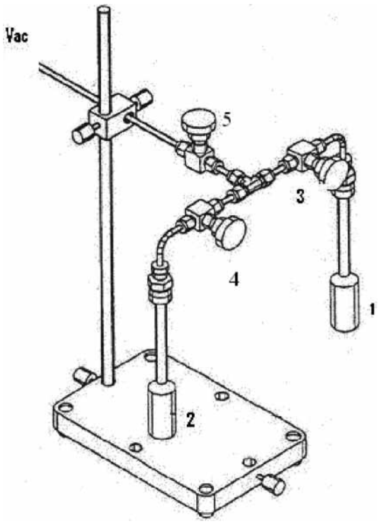

Figure 1. Apparatus for adsorption of pure solvent onto activated carbon.

PAHs were constructed. In a series of Erlenmeyer flasks (100 ml capacity), a known weight (0.25 ± .001 g) of the solid adsorbent was added to 50 ml of the PAHs compound at a concentration ranging from 50 to 1,000 mg/l in the appropriate organic solvent. The flasks were shaken in a thermostated water bath shaker kept at 25 ± 0.1°C for 48 h. The adsorbent particles were separated from the solution by filtration and the equilibrium concentration of the required PAHs was determined spectrophotometrically at $\lambda_{\max}$. The amount of the PAH adsorbed on the activated carbon was finally calculated employing the equation:

$$q_e = (C_o - C_e) \times \frac{V}{W} \tag{1}$$

where, q_e is the amount of PAHs adsorbed at equilibrium (mg/g); C_o and C_e are the initial and equilibrium concentrations of PAHs (mg/l), respectively; V is the volume of the sample in liter; and W is the weight of the adsorbent in grams.

Determination of Adsorption Capacity

Semi-quantitative tests were performed separately for the adsorption of each solvent as a single component from a gaseous state. A known mass of activated carbon was placed in a reservoir (1), and a suitable volume of a solvent was then placed in the other reservoir (2). The apparatus was evacuated for several minutes while the valve on the solvent line was closed, and the evacuation continued in the activated carbon line. After 30 minutes, the valve from the evacuation line to the pump was closed and the activated carbon line went in a vacuum. The adsorption started slowly through the opened valve on the solvent line (4) and left for several days to reach equilibrium. After equilibrium, the activated carbon sample was reweighed and the increase in the adsorbent weight was then calculated. The amount of solvent adsorbed at room temperature was then calculated.

RESULTS AND DISCUSSION

It is well known that the adsorption of PAHs onto porous solids is complicated by the structure of the PAHs molecule and the energetically heterogeneous nature of the solid surface. Thus, the surface area available in the geometrical sense, to the large molecule may contain a range of adsorption sites of different adsorptive power. The adsorption phenomenon is a manifestation of complicated interactions among the adsorbent, the adsorbate, and the solvent involved. The affinity between the adsorbent and the adsorbate is the main factor controlling the adsorption process (26). Thus, the influence

Table 3. Adsorption capacities of CAC towards pure solvents and pyrene

Solvent	Polarity	Dielectric constant	Solvent adsorbed mg/g	Pyrene adsorbed mg/g
Methanol	Polar (aliphatic)	32.63	234.21	182.84
Hexane	Non-polar (aliphatic)	1.90	240.40	165.93
Cyclohexane	Non-polar (cyclic)	2.00	281.70	151.14
Heptane	Non-polar (aliphatic)	1.90	293.71	149.03
Benzene	Non-polar (aromatic)	2.30	346.97	65.08

of the chemical structure of the adsorbate and the polarity of the non-aqueous solvent has been critically investigated.

A preliminary investigation of the adsorption capacities of the CAC towards pure organic solvents are summarized in Table 3. The data revealed that the aromatic solvent (benzene) adsorbs to CAC better than the polar solvent (methanol). Furthermore, the adsorption of pure solvents with the same number of carbons (i.e. n-hexane, cyclohexane, and benzene) is dependent of the chemical structure of the solvent. The high affinity of CAC towards aromatic solvent can be attributed to its aromatic nature rather than the solvent polarity where the dielectric constant slightly increases from aliphatic to aromatic hydrocarbons.

The adsorption capacities of CAC towards PY from non-aqueous solvents are also, listed in Table 3. The data reveals that the chemical structure of the solvent plays an important role in the adsorption process. It can be seen that the adsorption of PY mixed with polar or non-aromatic solvents is higher than that mixed with the aromatic solvent. The high affinity of a CAC towards an aromatic hydrocarbon solvent creates a competition between the solvent and PAHs molecules as a result of the aromaticity of these molecules. Thus, the aromatic solvent molecules are able to compete with PY molecules and reduce the adsorption uptake to one-third of its uptake from methanol. Due to the aromatic nature of benzene molecule, a $\pi-\pi$ complex will be formed between the π- electrons of the benzene rings and the active sites on the carbon surface (27). The small molecular size of the benzene molecule, as compared to PY molecular size, enhances the adsorption of benzene by diffusion to narrow pores which are inaccessible for the large PY molecule. A little competition between the non-aromatic hydrocarbon solvents and PY molecules has been noticed. This explains why activated carbon has lower adsorption capacities for PY in an aromatic solvent than in cyclic and aliphatic hydrocarbon solvents.

Figures 2–4 clearly shows the adsorption capacity of a CAC towards PAHs in organic solvents is a function of PAHs structure and solvent polarity. In other words, PY (four fused aromatic rings) adsorbs to CAC better than NA and AN (two and three fused aromatic rings, respectively). Additionally, the adsorption of PY in methanol is higher than that in

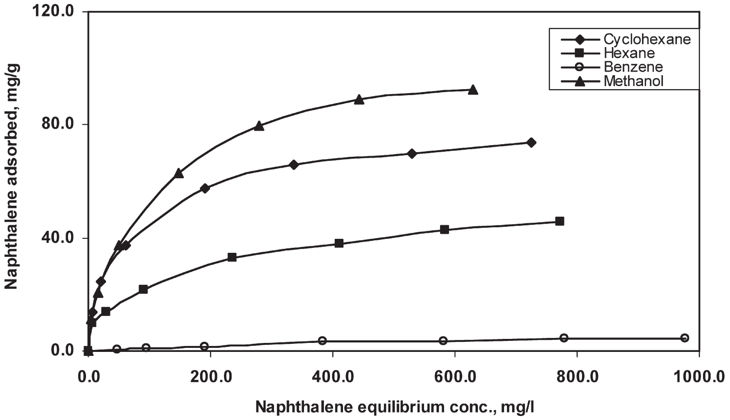

Figure 2. Adsorption isotherms of naphthalene onto CAC from organic solvent at 25 ± 0.1°C.

non-polar hydrocarbons solvents. This behavior is most likely attributed to the difference in the polarity of methanol and hydrocarbon solvents. The low solubility of PAHs in a polar solvent and their high affinity to the non-polar surface of the activated carbon may represent an important parameter for increasing the adsorption capacity from methanol.

The high affinities between PAHs and benzene as a solvent (solubility) restrict the adsorption of PAHs molecules in the aromatic solvent (28). The solubility of a substance decreases as the difference between its polarity and the polarity of the solvent increases. Thus, the adsorbed amount of the PAH molecule increases as its solubility in the solvent decreases.

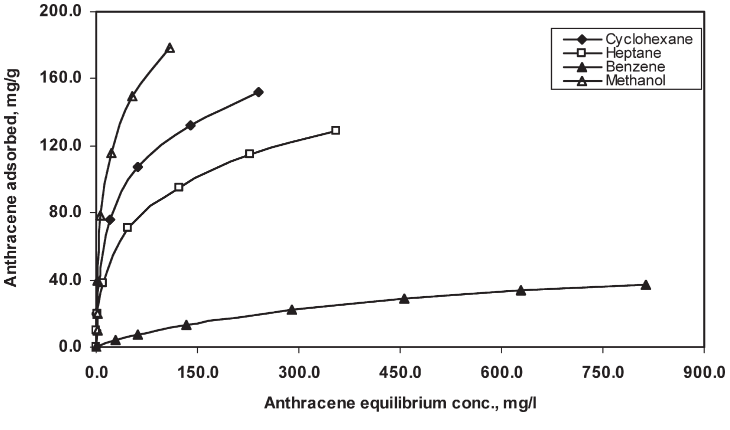

Figure 3. Adsorption isotherms of anthracene onto CAC from organic solvent at 25 ± 0.1°C.

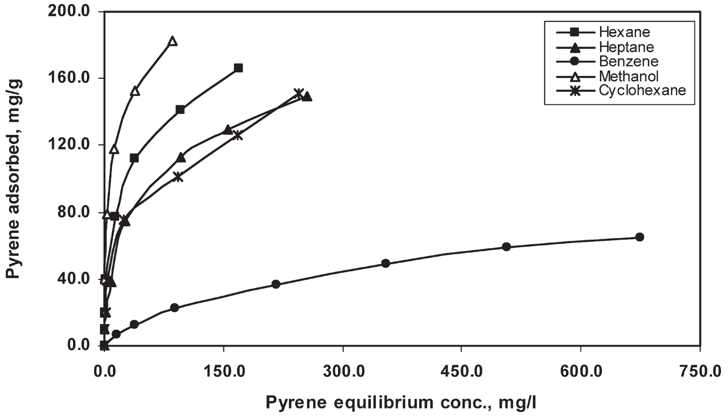

Figure 4. Adsorption isotherms of pyrene onto CAC from organic solvent at 25 ± 0.1°C.

Figure 5 shows the effect of the PAH's molecular weight on the adsorption process. An increase in the number of aromatic rings in a PAH molecule decreases its solubility and as a result its adsorption ability to activated carbon increases (29–33).

Table 4 reflected the influence of the solubility of the PAHs on their adsorption ability to activated carbon. The maximum adsorption capacity for the tested PAHs was obtained in methanol due to the lowest affinity between the PAHs molecules and the solvent molecules. The PAHs have low solubility in methanol due to incompatible intermolecular forces while the PAHs have a high solubility and great interaction forces towards benzene molecules, yielding a minimum adsorption capacity in an aromatic

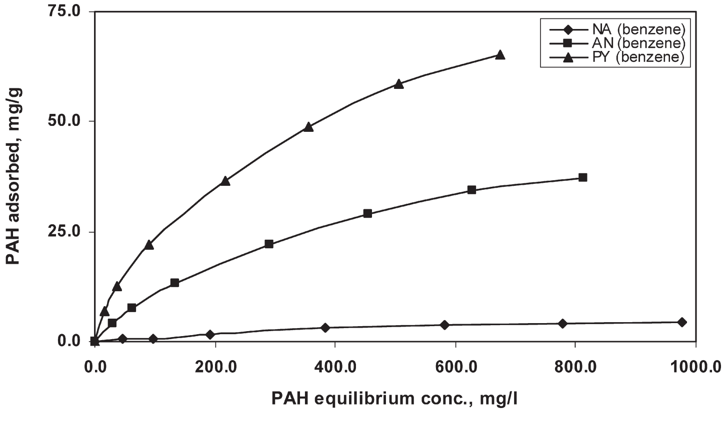

Figure 5. Adsorption isotherms of the tested PAHs in benzene at 25 ± 0.1°C.

Table 4. Adsorption capacities of CAC towards some PAHs in different solvents

	Solvent				
PAHs	Methanol mg/g	Hexane mg/g	Heptane mg/g	Cyclohexane mg/g	Benzene mg/g
Naphthalene	92.62	45.50	53.47	75.97	4.40
Anthracene	178.15	—	129.04	151.93	37.30
Pyrene	182.84	165.93	149.03	151.14	65.08

solvent. The adsorption uptake follow the order:

Methanol $>$ n-hexane $>$ cyclohexane

Increasing of the molecular weight of the aliphatic solvents (e.g., hexane and heptane) has little effect on the adsorption capacity of NA and PY (Fig. 6). It was noticed that, there is a slight increase in PY uptake in hexane than in heptane which can be attributed to the lower solubility of PY in hexane. Furthermore, the adsorption of PAH from cyclic hydrocarbon solvents (e.g., cyclohexane) increases is high compared to PAHs from aliphatic hydrocarbon solvents (Fig. 7).

The adsorption isotherms of the tested PAHs in different organic solvents using CAC at 25 ± 0.1°C are demonstrated in Figs. 2–4. The adsorption of PAHs showed isotherms close to type I (34). The initial part of the isotherm represents the micropore filling. The slope of the plateau at higher concentrations is due to multilayer adsorption on the non-microporous surface (mesopores, macropores, and the external surface) (23).

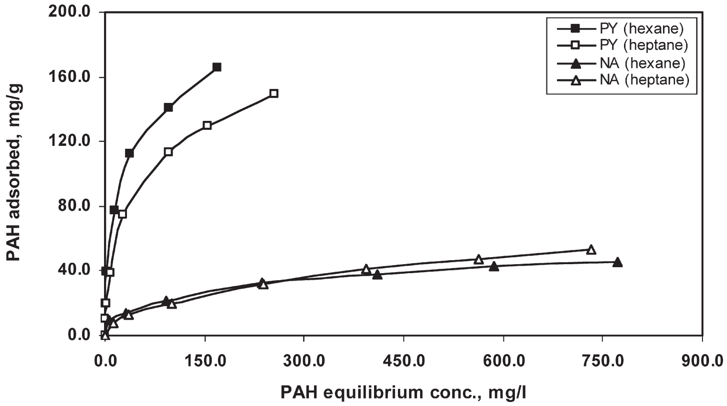

Figure 6. Adsorption of naphthalene and pyrene from hexane and heptane at 25 ± 0.1°C.

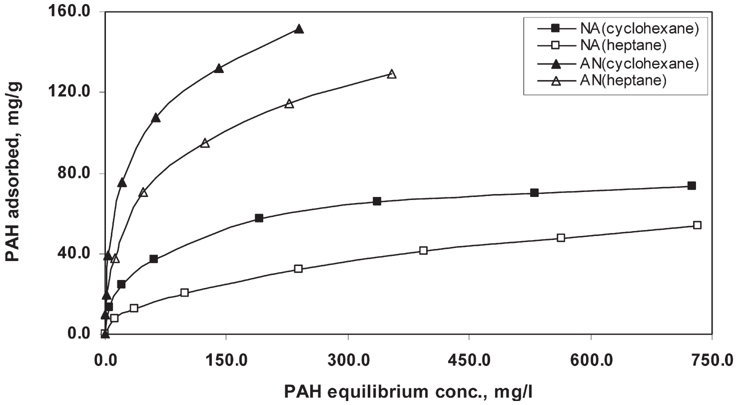

Figure 7. Adsorption of naphthalene and anthracene from cyclohexane and heptane at 25 ± 0.1°C.

The Langmuir and Freundlich models were employed for modeling the adsorption isotherms (35). The linear forms for Langmuir and Freundlich models are as shown in Eqs. (2–3), respectively:

$$\frac{C_e}{q_e} = \frac{1}{K_L} + \frac{a_L}{K_L} C_e \tag{2}$$

$$\log q_e = \log K_F + \frac{1}{n} \log C_e \tag{3}$$

where, q_e is the adsorption capacity at equilibrium (mg/g); C_e is the adsorbate equilibrium concentration mg/l, K_L and a_L are the Langmuir constants (l/mg), K_F and n are the Freundlich constants.

The data suggest the applicability of the Langmuir isotherm for the present systems, and formation of monolayer coverage of the adsorbate at the outer surface of the adsorbent. Langmuir constants and correlation coefficient (R^2) in organic solvents are summarized in Table 5. The R_L values for all studied systems were found in the range $0 < R_L < 1$ suggesting favorable adsorption of PAHs onto the investigated adsorbent.

The parameters K_F and n of the linear plots using Freundlich equation are reported in Table 6. The n values for all studied systems, range between 1.320 and 2.892. The minimum value being for adsorption of PAHs from benzene while the maximum value is for adsorption from methanol. The results are in good agreement with the data reported in literature (36). Table 6 shows that the highest KF values obtained are for adsorption of PAHs from methanol systems while the lowest values are for the adsorption of PAHs from benzene solvent.

Table 5. Langmuir parameters for PAHs adsorption from different solvents

PAHs	Langmuir parameters					
	Solvent	K_L dm^3 g^{-1}	a_L dm^3 mg^{-1}	q_{max}, mg g^{-1}	R_L (−)	R^2
Naphthalene	Methanol	1.428	0.014	102.35	0.067	0.99
	Cyclohexane	1.493	0.019	77.630	0.049	0.986
	Hexane	0.573	0.012	48.890	0.079	0.959
	Heptane	0.383	0.006	61.519	0.138	0.970
	Benzene	0.011	0.001	8.047	0.422	0.980
Anthracene	Methanol	14.201	0.072	197.72	0.014	0.983
	Cyclohexane	10.774	0.070	154.830	0.014	0.971
	Heptane	5.041	0.038	132.803	0.0257	0.970
	Benzene	0.139	0.003	54.978	0.284	0.998
Pyrene	Methanol	37.749	0.200	188.90	0.005	0.985
	Cyclohexane	8.889	0.060	149.22	0.017	0.950
	Hexane	16.307	0.097	168.676	0.010	0.964
	Heptane	7.055	0.045	155.172	0.022	0.982
	Benzene	0.364	0.004	84.988	0.189	0.991

Table 6. Freundlich parameters for PAHs adsorption from different solvents

	Freundlich parameters			
PAHs	Solvent	K_F (dm^3 g^{-1})	n	R^2
Naphthalene	Methanol	5.666	2.185	0.962
	Cyclohexane	8.013	2.822	0.965
	Hexane	4.669	2.892	0.994
	Heptane	2.314	2.095	0.999
Anthracene	Benzene	0.028	1.320	0.938
	Methanol	17.675	1.816	0.916
	Cyclohexane	19.905	2.536	0.981
	Heptane	13.830	2.527	0.989
Pyrene	Benzene	0.475	1.502	0.988
	Methanol	31.091	2.174	0.928
	Cyclohexane	18.754	2.579	0.988
	Hexane	26.106	2.649	0.988
	Heptane	14.657	2.278	0.980
	Benzene	1.376	1.657	0.993

CONCLUSION

The adsorption of PAHs onto non-polar adsorbents such as activated carbon depends on the chemical structure of the solvent and the solvent aromaticity. The adsorption capacity of the PAHs on CAC from organic solvents containing aromatic ring decreases due to the competition between the aromatic solvent and PAHs molecules. The polarity of media has a significant effect on the adsorption capacity of the adsorbent. The low solubility of the PAHs in polar solvents increases the adsorption uptake due to the lower affinity between PAHs and polar media. The adsorption isotherms of the tested PAHs can be predicted successfully using Langmuir and Freundlich isotherms with good correlation coefficients.

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