



Performance of butyl rubber–based macroporous sorbents as passive samplers

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Received: 31 December 2019 / Accepted: 17 April 2020 / Published online: 28 April 2020
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Abstract

In this study, two macroporous butyl rubber (BR)–based sorbents prepared in benzene (H-BR) and in cyclohexane (L-BR) with different porosities were synthesized by cryogelation technique. Their performances as a passive sampler were studied and then compared with commercially available silicon rubber (polydimethylsiloxane, PDMS) passive sampler. For that aim, polycyclic aromatic hydrocarbon (PAH) absorption rates of the sorbents in the short-term and their accumulation capacities in the long-term periods were investigated. Four PAHs (naphthalene, phenanthrene, fluoranthene, and pyrene) with a different number of aromatic rings were utilized. The concentrations of the PAHs in solutions were quantified by fluorescence spectrophotometer. The results showed that the BR sampler prepared in benzene (H-BR) generally has the highest absorption rates for all PAHs. The rate constants k (h^{-1}) of the H-BR, L-BR, and PDMS samplers were found as 1.07, 0.55, and 0.55 for naphthalene; 0.73, 0.16, and 0.09 for phenanthrene; 0.24, 0.26, and 0.08 for fluoranthene; and 0.97, 0.38, and 0.17 for pyrene, respectively. The highest PAH absorption capacity was found for the BR sorbents prepared in benzene for all PAHs. Thus, benzene was selected as the organic solvent rather than cyclohexane for further studies in the preparation of butyl rubber–based samplers. The H-BR possessing the highest absorption rate and capacity underlines their usage as a capable passive sampler for both short- and long-term monitoring activities in the aquatic environments.

Keywords Passive samplers · Butyl rubber sorbents · PAH · Organic pollutants · Marine monitoring · Absorption kinetics

Introduction

Polycyclic aromatic hydrocarbons (PAHs) are organic chemicals having different structures. They are considered as ubiquitous chemical compounds observed in the atmosphere, soil, and different aquatic matrices (sediment, biota, and water). They bioaccumulate and have toxic, mutagenic, and

carcinogenic effects on living organisms in the aquatic ecosystem (Kumar et al. 2016). They also have significant health risks for human health (Kallenborn et al. 2012; Venkatesan and Halden 2014; Rengarajan et al. 2015). PAHs can be classified according to their origins as pyrogenic, petrogenic, and biological in natural environments. Pyrogenic PAHs are the products from incomplete combustion of organic substances such as wood and coal, while petrogenic PAHs are originated during the manufacturing and processing of the petroleum products and also from the oil spills caused by ship accidents, maritime traffic, oil platforms, and underwater pipes (Sciaccia and Conti 2009; Abdel-Shafy and Mansour 2016; Pogorzelec and Piekarska 2018).

Passive samplers are materials that are commonly used for the detection and monitoring of the organic compounds with high hydrophobicity in aqueous media. Since these compounds including polychlorinated biphenyls (PCBs), PAHs, and organochlorine pesticides like DDT have very low water solubilities, it is hard to detect their existence and concentrations in water. Moreover, conventional sampling methods are

Responsible editor: Tito Roberto Cadaval Jr

Electronic supplementary material The online version of this article (<https://doi.org/10.1007/s11356-020-08945-4>) contains supplementary material, which is available to authorized users.

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incapable of detecting the freely dissolved concentrations (C_{free}) of hydrophobic chemicals which directly govern the toxicity of the pollutants for the organisms (Reichenberg and Mayer 2006; Smedes et al. 2010). These methods detect total concentrations of the pollutants (C_{total}) including freely dissolved pollutants and pollutants bounded to suspended materials (Booij et al. 2016). Moreover, due to the lipophilic characteristics of organic pollutants, organisms are also suitable tools for biomonitoring activities. Organic substances are accumulated in the tissues of species, and their water concentrations can be determined using the bioconcentration factor (BCF) (Arnot and Gobas 2006). Phytoplankton (Hosmani 2014), fish (Gassel et al. 2013), and mussels (Kasiotis et al. 2015) have been commonly used to monitor the concentrations and the effects of the pollutants. However, field studies with living organisms have several drawbacks. Mobility of the organisms makes it hard to monitor certain areas, and because of the metabolic activities, determination of the pollutant concentrations is a challenging task; besides, high concentrations may result in mortality of the organisms (Holt and Miller 2010). Similar to the conventional water sampling methods, organisms also accumulate the suspended particulate materials in their tissues; therefore, instead of freely dissolved concentration, total concentrations of the chemicals were determined. When the disadvantages of the bioindicator species during the monitoring studies are considered, passive samplers seem more applicable tools in aquatic environments.

Various passive sampler devices (PSDs) have been developed including semipermeable membrane devices (SPMDs) (Huckins et al. 1990), solid-phase microextraction devices (SPME) (Arthur and Pawliszyn 1990), silicon rubber passive samplers (polydimethylsiloxane, PDMS) (Smedes 2007), low-density polyethylene samplers (LDPE) (Adams et al. 2007), and polyoxymethylene (POM) (Cornelissen et al. 2008). Since then, enormous progress has been made to improve samplers' performances. Recently, developing selective novel passive samplers is a focused topic. Several passive samplers have been developed for different purposes such as detection of the heavy metals (Dou et al. 2019), micropollutants (Moschet et al. 2015; Mutzner et al. 2019), and per- and polyfluoroalkyl substances (PFASs) (Kaserzon et al. 2019). Even though various types of passive samplers have been developed, semipermeable membrane devices (SPMDs) are the only standardized and commercialized samplers (Huckins et al. 2006; Booij and Smedes 2010).

Monitoring organic contaminants in the marine and freshwater environment is regulated by Water Frame Directive (WFD) (European Commission 2009) in Europe and by the Clean Water Act (CWA) in the USA. In WFD, the primary method for monitoring activities is the batch sampling of surface water, sediment, and biota; however, passive samplers are considered as complementary tools which can support or contradict the batch sampling results. On the other hand, the use

of passive sampling devices is actively encouraged and supported by the US Environmental Protection Agency (EPA) (EPA 2012). Moreover, the Oslo-Paris Convention for the protection of the marine environment of the North-East Atlantic (OSPAR) indicates the drawbacks of the conventional sampling methods and emphasizes the advantages of the passive samplers to determine the time-averaged concentrations of the pollutants (OSPAR Commission, 2013). Passive samplers may be monophasic or bi-phasic. Monophasic passive samplers were strongly suggested due to the practical reasons such as preparation, use, and analysis.

Macroporous, monophasic butyl rubber (BR) sorbents were prepared from commercially available BR via cryogelation technique under different conditions and specifically in different organic solvents leading to rubber materials with different porosities and morphologies (Ceylan et al. 2009; Muslumova et al. 2019). The effect of these parameters on the performance of BR passive samplers has not been reported before. Moreover, although the performance of BR sorbents for the removal of crude oil and PAHs from seawater was investigated (Ceylan et al. 2009; Yilmaz et al. 2014; Yetiskin et al. 2019), the absorption rates and capacities of BR samplers to be used as passive samplers have not been investigated before.

In the present study, PAH sampling performances of BR-based passive samplers with two different porosities were investigated. As a reference, a conventional and widely used passive sampler, namely polydimethylsiloxane (PDMS), was also used in the experiments. PAH absorption rates of passive samplers in the short term and absorption capacities in the long term were determined. Four different PAHs (naphthalene, phenanthrene, fluoranthene, and pyrene) with a different number of aromatic rings were selected. As will be seen below, the absorption rates and capacities of BR samplers are much higher than those of the conventional PDMS passive sampler. It was also found that both the porosity and pore morphology of BR sampler have a significant effect on their performances.

Materials and methods

Materials

Butyl rubber (BR) passive samplers were prepared by the cryogelation technique conducted at $-18\text{ }^{\circ}\text{C}$ as reported before (Muslumova et al. 2019). Briefly, BR (5 g) was dissolved in 100 mL of cyclohexane or benzene at $23 \pm 2\text{ }^{\circ}\text{C}$ overnight to obtain a homogeneous 5% w/v BR solution. After the addition of 0.3 mL of sulfur monochloride, S_2Cl_2 , as a cross-linker and stirring, the solution was transferred into several plastic syringes and petri dishes of 14 mm and 10 cm in diameters, respectively. Finally, syringes and petri dishes were

placed in a thermostat at $-18\text{ }^{\circ}\text{C}$ to conduct the cryogelation reactions for 24 h. After purifying and drying, BR passive samplers in the form of cylinders and sheets were obtained. The total pore volume V_p and the total porosity P of BR passive samples were determined from the methanol uptake and equilibrium swelling ratios in toluene, respectively, as detailed previously (Yetiskin et al., 2019). The passive samplers prepared in benzene (H-BR) and cyclohexane (L-BR) have porosities and total pore volumes of 85% and 7.9 mL g^{-1} , and 80% and 3.5 mL g^{-1} , respectively, where H and L stand for high and low porosities. The morphology of BR passive samplers was studied using scanning electron microscopy (SEM, Jeol JSM 6335F field emission SEM) after coating with gold using a sputter coater. The average diameter of the pores was calculated by analyzing at least 10 SEM images of various magnifications using ImageJ image processing software (US National Institutes of Health, Bethesda, MD). Commercial PDMS elastomer (kSil GP60) used as a reference passive sampler was provided from Silicone Engineering Ltd. (Blackburn, UK). PDMS samplers were cleaned before the experiment in the Soxhlet extractor with ethyl acetate for 5 days. SEM measurements of PDMS samplers were conducted on a Tescan GAIA 3 field emission SEM after sputter-coating with gold palladium. PAHs utilized in the absorption tests were obtained from Sigma-Aldrich (naphthalene) and Fluka (phenanthrene, fluoranthene, and pyrene).

Analysis of PAH compounds

PAH concentrations of the solutions were determined using a fluorescence spectrophotometer (Perkin Elmer, LS 55). The excitation and the emission wavelengths of each PAH chemicals were determined (Table 1) and PAH concentrations were measured against calibration standards.

Determination of absorption rates and absorption capacities

Two experimental systems have been designed to determine the absorption rates and capacities of the passive samplers. In the batch system, L-BR, H-BR, and PDMS samplers were cut into 0.100-g pieces and placed into 40-mL glass vials, which

were filled with the aqueous solutions of the PAHs under their solubility limits. The initial concentrations of PAH solutions prepared with filtered (active carbon and $0.45\text{ }\mu\text{m}$ cellulose membrane) natural seawater (22 ppt) are given in Table 2. The vials were then placed onto a horizontal shaker (IKA, HS-260) and stirred at 120 rpm in a temperature-controlled room ($21 \pm 0.5\text{ }^{\circ}\text{C}$). The concentrations of the PAH solutions were determined by sub-sampling after 0.5, 2, 5, 8, and 24 h, and the data were used to calculate the absorption rates. Moreover, the PAH absorption capacities were quantified in a semi-batch system by renewing the PAH solutions daily. The concentrations of PAHs were measured every 24 h for a time period of 12 days.

Results and discussions

The BR passive sampler denoted as H-BR has a relatively high porosity and pore volume as compared with L-BR. Moreover, the pore morphologies of H-BR and L-BR samplers show significant differences, as seen in the SEM images in Fig. 1a and b, respectively. H-BR has a dual-scale porosity consisting of large pores of $1.6 \pm 0.8\text{ mm}$ in sizes together with $45 \pm 15\text{ }\mu\text{m}$ -sized small ones. In contrast, L-BR has more uniform pores of $40 \pm 9\text{ }\mu\text{m}$ in diameter. To compare their performances as passive samplers, the commercial PDMS was also used in the PAH absorption experiments. SEM images of PDMS revealed the absence of pores in micrometer or nanometer size (Fig. 1c).

Absorption rates of the passive samplers

The variation of PAH concentrations in the filtered seawater containing H-BR, L-BR, and PDMS passive samplers was monitored by the fluorescence spectrophotometer. Figure 2 shows the results of the absorption behavior of the three passive samplers for four different PAH compounds.

In the figure, the absorbed amounts of PAHs are plotted against the contact time. Compared with PDMS, both BR passive samplers exhibit higher absorption performances. Moreover, the initial slopes of the absorption curves calculated covering the first 5-h data reveal fast absorption rates of BR sorbents as compared with PDMS. The results also show that H-BR, which has larger pores, a higher porosity, and pore volume, has a more rapid absorption rate than that of the L-BR. Results indicated that H-BR samplers have reached the equilibrium phase in 5 h for naphthalene and pyrene, in 8 h for phenanthrene. Unfortunately, the sub-sampling data for the 8 h of fluoranthene is missing due to the analytical problems. Therefore, the equilibrium time for the fluoranthene cannot be estimated from the data precisely. The superiority of the H-BR sampler over L-BR sampler can be explained with the fact that the H-BR sorbent has dual-scale porosity with large and small

Table 1 Excitation and emission wavelengths of the chemicals

Chemicals	Excitation wavelength (nm)	Emission wavelength (nm)
Naphthalene	224	330
Phenanthrene	252	370
Fluoranthene	252	402
Pyrene	238	398

Table 2 Water solubilities and initial concentrations of the selected PAHs

Chemicals	Water solubility (25 °C) ($\mu\text{g L}^{-1}$)	Initial concentration ($\mu\text{g L}^{-1}$)
Naphthalene	31,600	15,000
Phenanthrene	1150	1000
Fluoranthene	265	250
Pyrene	134	100

pores, a higher total porosity P , and the pore volume V_p . After 24 h, all the sorbents have absorbed approximately 80–90% of the initial PAH concentrations. Absorption rate constants of the samplers were calculated by using the PAH concentration data and first-order kinetics using Eq. (1);

$$C_t = C_{eq} (1 - e^{-kt}) \tag{1}$$

where C_t and C_{eq} are the absorbed amounts of PAHs in percentages at time t , and the equilibrium, respectively, and k is the rate constant. Comparative results for rate constant can be observed in Fig. 3.

Second-order kinetics was also applied to the data. Figure S1, as an example, was given in Online Resource 1 which shows the curve fitting results from the first- and second-order kinetics, and also the rate constants obtained from the fitting equations. Second-order kinetics resulted in very similar results; however, first-order fitting was slightly better. Rate constants of the H-BR samplers for the

naphthalene, phenanthrene, and pyrene have the highest values among all samplers, while the rate constants of H-BR and L-BR were very close to each other for the fluoranthene. The missing data for the fluoranthene probably result in under- or overestimation of k values for the whole experimental period. However, the calculated initial rate constants for all sorbents justify the highest performance of H-BR sorbents. The overall performance of the samplers for all PAHs can be represented by the average rate constants, which were found as 0.226 h^{-1} , 0.740 h^{-1} , and 0.357 h^{-1} for PDMS, H-BR, and L-BR samplers, respectively. These results clearly indicate that the macroporous structure of the BR samplers resulted in more rapid absorption of the PAHs when they are compared with the commercialized nonporous PDMS sampler. Much higher effectivity of BR sorbents as compared with PDMS is likely due to the fast absorption of PAHs in their hydrophobic inter-connected pore walls by diffusion as well as convection (Yetiskin et al. 2019). On the other hand, the PAH accumulation on the nonporous PDMS sampler (Fig. 1c) only occurs by

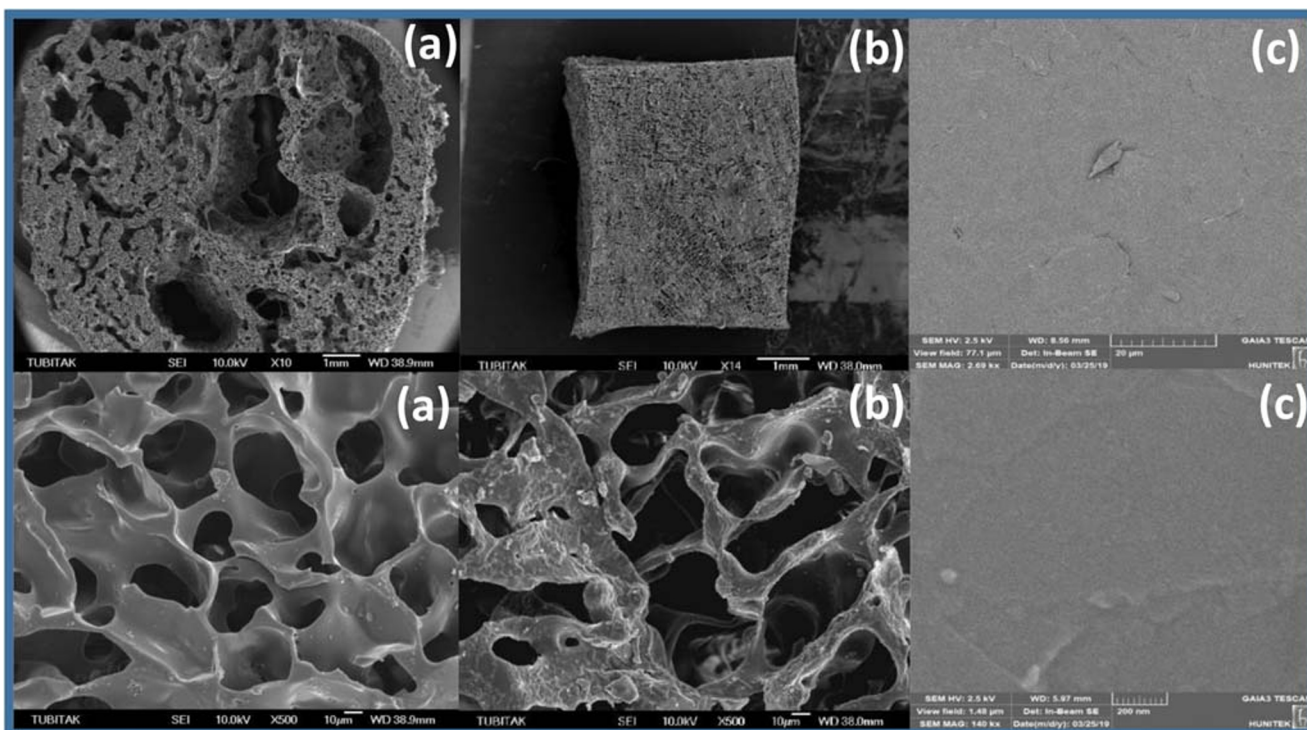
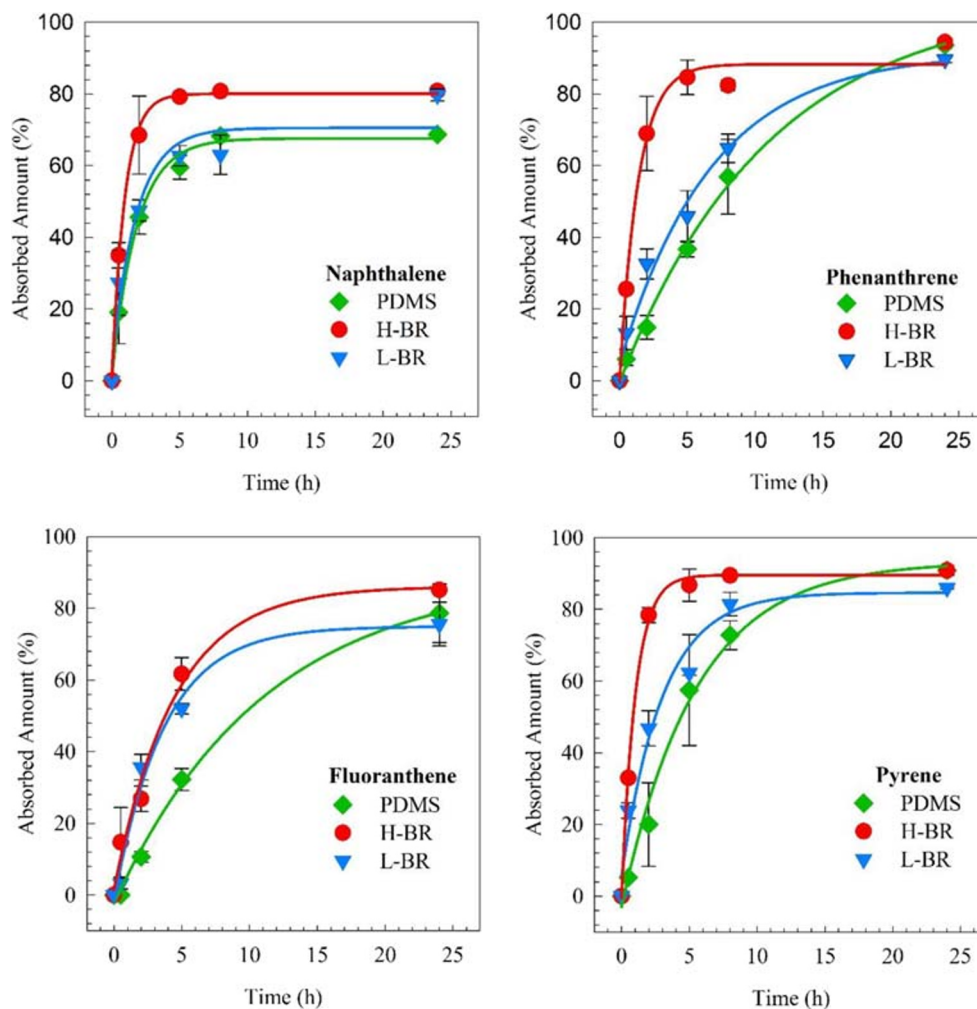


Fig. 1 SEM images of H-BR (a), L-BR (b), and PDMS passive samplers (c). Scale bars in the upper and bottom panels are 1 mm and 10 μm for both H-BR and L-BR, and 20 μm and 200 nm for PDMS, respectively

Fig. 2 Absorption data of passive samplers for 24 h (symbols) and regression curves, according to Eq. (1)



the slow diffusion mechanism. However, the selection of the organic solvent for BR sampler preparation has considerable effects on the sampling performances of the sorbents. The higher total porosity P and the pore volume V_p result in more

prompt absorption of the PAHs which are provided by the organic solvent benzene rather than cyclohexane.

Absorption capacities of the passive samplers

Absorption capacities of the samplers determined for 12 days with a semi-batch experimental system are presented in Fig. 4.

The figure shows that the H-BR sampler has the highest capacity over the other samplers for all PAHs. Because of the lower solubilities of the fluoranthene, pyrene, and phenanthrene compared with naphthalene, the equilibrium conditions have not been reached; therefore, the amount of absorbed PAHs in equilibrium (W_{eq}) cannot be determined experimentally. However, for naphthalene, which has the highest water solubility, the equilibrium state was reached in 6 days for PDMS sampler and in almost 12 days for porous H-BR and L-BR samplers. Absorbed amounts of naphthalene in the equilibrium phase for all passive samplers are presented in Fig. 5.

Absorbed amounts of naphthalene in the equilibrium phase by 1 g of samplers are 12 mg, 25 mg, and 28 mg for PDMS, L-

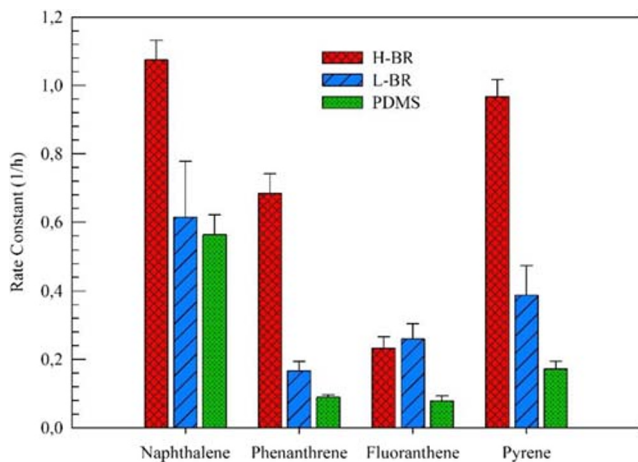
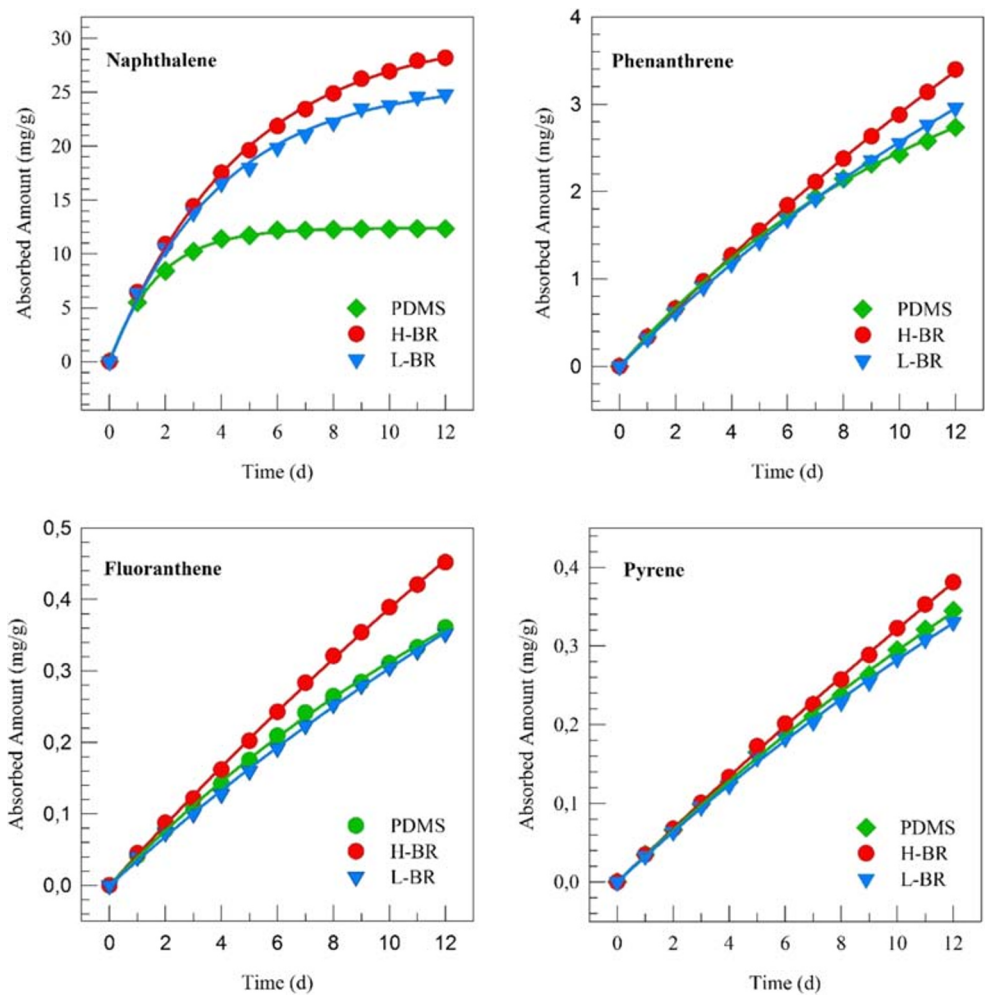


Fig. 3 Rate constants of the passive samplers for each PAH

Fig. 4 Cumulative absorbed amounts of PAHs by passive samplers (mg/g) for 12 days, experimental data (symbols), and regression curves



BR, and H-BR samplers, respectively. W_{eq} values obtained from the experimental results indicated that the absorption capacity of the BR samplers doubled the capacity of the PDMS sampler for naphthalene. On the other hand, H-BR

sorbents have superiority over L-BR sorbents in terms of naphthalene absorption capacities.

Since the equilibrium could not be reached for phenanthrene, fluoranthene, and pyrene in the experimental system,

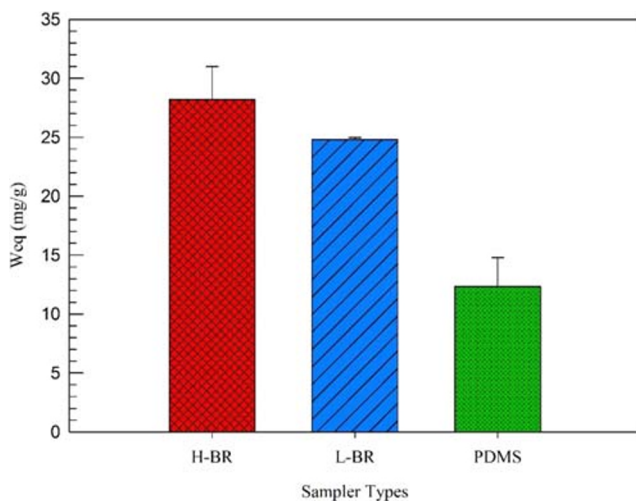


Fig. 5 Absorbed amount of naphthalene in equilibrium phase for all passive samplers (mg/g)

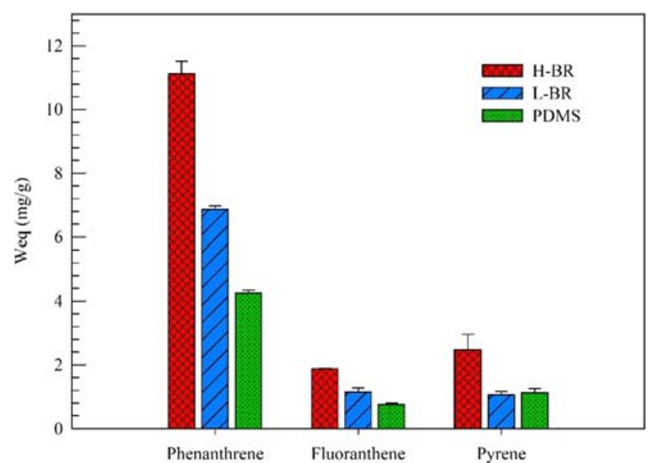


Fig. 6 The calculated amount of PAHs in equilibrium phases according to Eq. (1)

the absorption capacities were calculated from Eq. (1). The model results are given in Fig. 6.

It is seen that the H-BR sampler also has the highest absorption performance for these three PAHs. These results can be attributed to the differences in total porosity, P , and the pore volume, V_p , between H-BR and L-BR samplers.

Conclusion

The use of butyl rubber sorbents, initially developed for the removal of oil spills in aquatic environments, was found promising as passive samplers in the previous studies. In the present study, PAH absorption rates and capacities of two different BR samplers synthesized in benzene (H-BR) and cyclohexane (L-BR) were investigated and compared with the commercialized sampler PDMS in short- (24 h) and long-term (12 days) experimental studies. Results indicated that BR samplers have better absorption performances both in rate and capacity segments relative to the PDMS sampler for the selected PAHs. These results can be attributed to the macroporous structure of the BR samplers, which allows the absorption of the chemicals not only by a diffusion process but also the ambient convection through pores. On the other hand, results showed that organic solvent selection during the preparation of BR sampler has a significant effect on the sampling performances. BR samplers synthesized in benzene have advanced performance than the samplers synthesized in cyclohexane. Because the main differences between H-BR and L-BR samplers are the total porosity and total volume, the results highlight that they both are effective parameters for the passive sampling process. Outcomes of the study signify that BR samplers are promising as passive samplers. Further studies to determine the partition coefficients needed for back-calculating the water concentrations of PAHs are going on.

Funding information This research was funded by the Scientific and Technical Research Council of Turkey (TUBITAK), CAYDAG, 117Y099.

Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest.

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