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Vortex and air assisted liquid–liquid microextraction as a sample preparation method for high-performed liquid chromatography determinations

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ABSTRACT

A novel, simple and sensitive method based on vortex and air assisted liquid–liquid microextraction (VAALLME) technique coupled with high-performance liquid chromatography (HPLC) has been developed for quantitative analysis of β-naphthol, naphthalene and anthracene as model analytes. Unlike the dispersive liquid–liquid microextraction (DLLME), dispersive solvent and centrifuging step were eliminated in proposed technique. In this technique, extraction solvent was dispersed into the aqueous sample solution by using vortex. Phase separation was achieved via motion of air bubbles from the bottom to top of the extraction tube, which promoted the analytes transfer into the supernatant organic phase. Influential parameters on the extraction efficiency such as type and volume of extraction solvent, salt type and its concentration, vortex and aeration times, and sample pH were evaluated and optimized. The calibration curves showed good linearity ($r^2 > 0.9947$) and precision (RSD < 5.0%) in the working concentration ranges. The limit of detection (LOD) for β-naphthol, naphthalene and anthracene were 10, 5.0 and 0.5 ng mL^{-1}, respectively. The recoveries were in the range of 97.0–102.0% with RSD values ranging from 2.2 to 5.2%.

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1. Introduction

Most samples are not suitable for direct introduction into analytical instruments. For this reason, the sample preparation procedure is an important step in an analytical study. However, selection of sample preparation procedure depends on the analytes properties, the matrix, concentration level of analytes in the sample, the analytical techniques to be employed and their capabilities [1]. In the last two decades many extraction approaches for minimizing the environmental pollution emphasis on reducing organic solvent consumption in the extraction process.

Solid-phase microextraction (SPME) is the first microextraction technique that used to extract the analytes from a solution or headspace of sample [2–4]. SPME is a solventless procedure, which has double role of clean-up and pre-concentration of interested analytes. Limitations of the SPME for quantitative analysis are include: (a) longer extraction time, (b) limited volume of extractant phase (fiber coating), (c) carryover and memory effects, (d) fragility of the fibers and (e) the limited lifetime of fibers that results to increase analysis cost.

Liquid–liquid extraction (LLE) is one of the most common sample preparation techniques. LLEs involving a few milliliters or less of extraction solvent are termed microscale liquid–liquid extraction (MLLE). The fundamentals of MLLE techniques are similar to LLE with advantages of simpler automation and less solvent consumption. In 1996, a new microextraction technique namely single-drop microextraction (SDME) was introduced simultaneously by Liu and Dasgupta [5] and Jeannot and Cantwell [6].

Later, other types of liquid–liquid microextraction (LLME) techniques such as headspace liquid-phase microextraction (HS-LPME) [7–9], hollow fiber liquid–liquid microextraction (HF-LLME) [10–12], vortex-assisted liquid–liquid microextraction (VALLME) [13–15] and salt-assisted liquid–liquid microextraction (SALLME) [16–18] were developed. These methods have many advantages such as reduction in sample and solvent quantity, high enrichment factor and clean-up step.

In 2006, Assadi and co-workers [19] developed a novel microextraction technique, termed dispersive liquid–liquid microextraction (DLLME), which is based on a ternary component solvent system like homogeneous liquid–liquid extraction (HLLE) and cloud point extraction (CPE). In this method, the extraction is performed by an interaction between the sample and a cloud of

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fine extractant drops after injection of an appropriate mixture of extraction and disperser solvents into the aqueous sample. After formation of cloudy solution, the surface area between extracting solvent and aqueous sample increases which lead to quick extraction. Therefore, the extraction time becomes very short.

The air-assisted solvent extraction (AASX) method was used in engineering processes to remove of metals and organic contaminants from wastewater. In this method, a solvent-coated bubble is used to contact between organic and aqueous phases [20,21]. Aeration causes the extraction solvent to form a thin layer on bubbles and leads to increases the contact area between two phases [22]. Due to increasing interfacial area between extraction solvent and aqueous phase, analytes can be extracted into the organic phase in short time with higher efficiency.

Recently, an air-assisted liquid–liquid microextraction method (AALLME) as a new version of DLLME was developed for extraction and preconcentration of phthalate esters in aqueous samples [23]. Due to elimination of disperser solvent in AALLME method, volume of extraction solvent was decreased in comparison with DLLME. In order to increase the contact between analytes and extraction solvent, the mixture of aqueous sample solution and extraction solvent was sucked and injected with a syringe for several times in a conical test tube.

In this work, a novel vortex and air assisted liquid–liquid microextraction (VAALLME) technique was developed for determination of trace levels of β-naphthol, naphthalene and anthracene in wastewater samples. Unlike DLLME method, extraction was performed without using disperser solvent and centrifuging step. After mixing of sample solution and extraction solvent by using vortex, the cloudy mixture was transfer to a long tube and subjected to aeration process. Aeration leads to phase separation and increases analytes transfer to organic phase. Finally, upper organic phase was removed and injected to high-performance liquid chromatography (HPLC) system. The influences of the different experimental parameters on the extraction efficiency of model analytes are studied and optimized.

2. Experimental

2.1. Chemicals and materials

Acetonitrile (HPLC grade), methanol, cyclohexane, octanol, 2-decanol, sodium carbonate, ammonium acetate, sodium chloride, sodium hydroxide and orthophosphoric acid were purchased from Merck Chemical Company (Darmstadt, Germany). Naphthalene, β-naphthol and anthracene were obtained from Sigma-Aldrich (USA). All solutions were prepared with deionized water from a Milli-Q system (Millipore, USA).

2.2. Chromatographic conditions

The HPLC system (Shimadzu Corporation, Kyoto, Japan) which consisted of a quaternary pump (LC-10ATvp), UV–vis detector (SPD-M10Avp), vacuum degasser and system controller (SCL-10Avp) was used. A manual injector with a 10 μL sample loop was applied for loading the sample. Class VP-LC workstation was employed to acquire and process chromatographic data. A reversed-phase C_{18} analytical column (Shim-Pack VP-ODS, 250 mm × 4.6 mm i.d., Shimadzu, Japan) was used.

The mobile phase consisted of water and acetonitrile (40:60, v/v). Prior to preparation of the mobile phase, water and acetonitrile were degassed separately using a Millipore vacuum pump. The UV detector was set at 254 nm. The chromatograms were run for 15 min at a flow rate of 1.0 mL min^{-1} at ambient temperature.

2.3. Sample preparation

Standard stock solutions were prepared by dissolving each analyte in methanol with concentration of 100 μg mL^{-1}. Working standard solutions at different concentrations were prepared freshly by mixing the appropriate volumes of the stock solutions and diluting with deionized water.

Wastewater samples were collected from Shazand Petrochemical Corporation (Arak, Iran). Samples were filtrated through a 0.45 μm PTFE membrane and were adjusted to the pH of 7.0 prior to extraction.

2.4. VAALLME procedure

10 mL of sample or standard solution was transferred into a 50 mL conical polypropylene centrifuge tube. 1 g of sodium carbonate and 500 μL of octanol/cyclohexan (50:50, v/v) as extraction solvent were added and then the mixture was vortexd (DRAGON LAB MX-S, Beijing, China) at 2500 rpm for 2 min. The cloudy mixture was transferred into a long glass tube and subjected to aeration process by using an air pump (model XP-2200A, China) until phase separation occurs and aqueous phase was clear. Then the organic phase was moved to the top of the tube by using water injection. Finally, 10 μL of organic phase was withdrawn and injected into the HPLC system for analysis. The schematic diagram of extraction process was illustrated in Fig. 1.

3. Results and discussion

Various parameters such as type and volume of extracting solvent, vortex and aeration times, salt type and its concentration and sample pH can be affected on extraction efficiency. The effects of these parameters on extraction were studied and optimized.

3.1. Selection of extraction solvent

In order to select suitable extraction solvent several parameters must be considered: (a) no interference with analytes signal, (b) high extraction efficiency for analytes, (c) low solubility in aqueous solution and (d) compatibility with detection system. Various extraction solvents such as octanol, 2-decanol, cyclohexane, and mixture of octanol/cyclohexane at different ratios were examined. The results were illustrated in Fig. 2. Low extraction efficiency of 2-decanol and octanol as extraction solvent can be attributed to high viscosity of these solvents, which decrease the diffusion coefficients of the analytes. In addition, polarity of extraction solvent is another important parameter. Therefore, different ratios of octanol and cyclohexane were used. According to results, mixture of octanol/cyclohexane (50:50 v/v) was selected as appropriate extraction solvent.

3.2. Volume of extraction solvent

Fig. 3 shows the influence of extraction solvent volume on the analytes extraction. It can be observed that the peak areas of analytes increased with increasing extraction solvent volume up to 200 μL and then decreased. In lower extraction solvent volumes (< 200 μL), extraction of analytes are not completed. In the other hand, enrichment factor (EF) decreases with increasing volume of the extraction solvent. Therefore, 200 μL was selected as the optimum volume in this study.

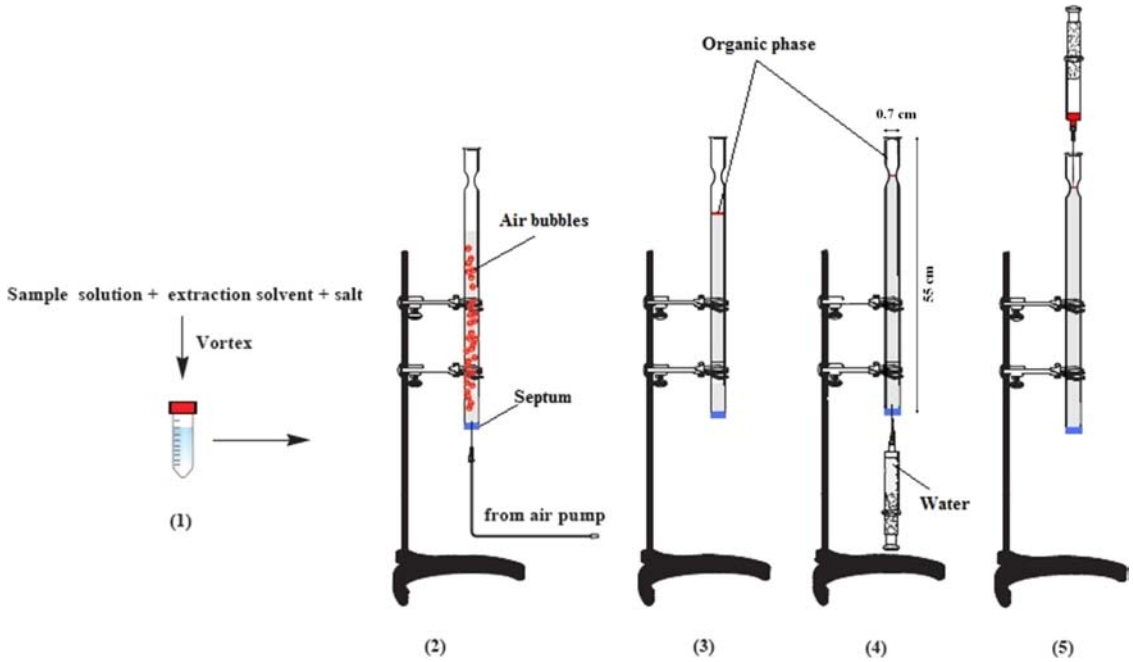

Fig. 1. Schematic diagram of proposed vortex and air-assisted liquid–liquid microextraction (VAALLME) procedure. (1) the mixture of sample solution, extraction solvent and salt was mixed using a vortex mixer, (2) the cloudy mixture was transferred to long tube and connected to air pipe from an air pump (3) separation of organic phase by using aeration process, (4) elevating the organic phase by using water injection through the septum in the bottom of tube, (5) removal of 10 μL of the collected organic phase in the narrow region of the tube for analysis.

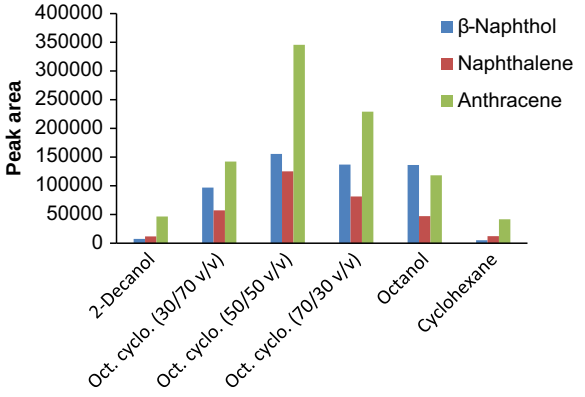

Fig. 2. Effect of extraction solvent on the analytes extraction (oct.; octanol, cyclo.; cyclohexane). Extraction conditions: extraction solvent volume, 200 μL; salt concentration, 10% w/v; vortex time, 2 min; aeration time; 5 min. Concentration of analytes were as follow: β-naphthol; 2.5, naphthalene; 2.5 and anthracene 0.125 μg mL^{-1}.

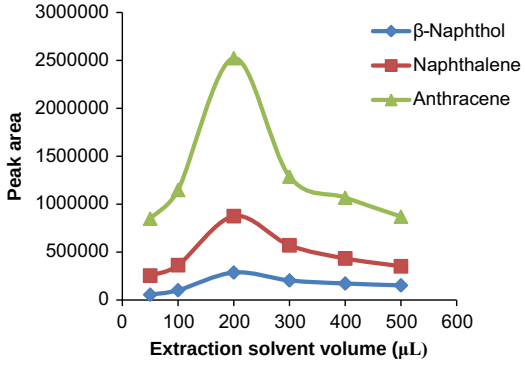

Fig. 3. Effect of extraction solvent volume on the analytes extraction. Extraction conditions: extraction solvent, octanol/cyclohexane (50:50 v/v); salt concentration, 10% w/v; vortex time, 2 min; aeration time; 5 min. Concentration of analytes were as follow: β-naphthol; 2.5, naphthalene; 2.5 and anthracene 0.125 μg mL^{-1}.

3.3. *Effect of vortex time*

Vortex was chosen to produce cloudy solution, which enhances the contact between the extraction solvent and aqueous sample solution. Therefore, vortex time as an important parameter should be optimized. Various experiments were performed by using different vortex times in the range of 30–150 s. The results are displayed in Fig. 4. The maximum peak areas were obtained at vortex time of 120 s. Hence, 120 s was chosen as the optimum vortex time.

3.4. *Effect of aeration time*

After the vortex step, sample solution becomes cloudy which indicates extraction solvent dispersed in the aqueous solution. In the proposed method, phase separation was performed by using

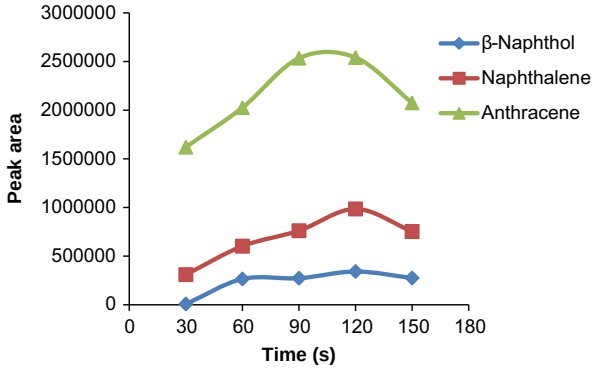

Fig. 4. Effect of vortex time on the analytes extraction. Extraction conditions: extraction solvent, octanol/cyclohexane (50:50 v/v); extraction solvent volume, 200 μL; salt concentration, 10% w/v; aeration time; 5 min. Concentration of analytes were as follow: β-naphthol; 2.5, naphthalene; 2.5 and anthracene 0.125 μg mL^{-1}.

aeration process instead of conventional centrifuging step. Aeration will be continuing until the sample solution becomes clear. The aeration process leads to phase separation and increases the extraction efficiency. It is assumed the air bubbles in sample solution containing the analyte molecules. When the bubbles reach to the sample surface (organic layer) and burst, the analytes molecules were transferred to the organic phase. The aeration time was studied in the range of 2 to 7 min. Phase separation was not observed in less than 2 min. With increasing the aeration time, extraction efficiency of analytes were increased up to 5 min and then level off (Fig. 5). Therefore, 5 min was selected as the optimum aeration time for further experiments.

3.5. Effect of salt type and its concentration

Generally, in LLE methods salt addition can lead to increase extraction efficiency. The solubility of analytes and extraction solvent in the aqueous phase decrease by salt addition and facilitated the analytes transfer into the organic phase. In this work, various salts including sodium chloride, ammonium acetate and sodium carbonate were used at different concentrations. Extraction efficiency with sodium carbonate was more than two other salts. Therefore, sodium carbonate concentration was investigated in the range of 0–12% w/v. The results in Fig. 6 indicate, extraction efficiency of the analytes increased up to 10% w/v

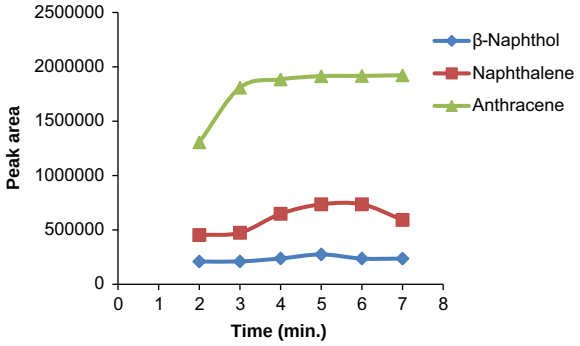

Fig. 5. Effect of aeration time on the analytes extraction. Extraction conditions: extraction solvent, octanol/cyclohexane (50:50 v/v); extraction solvent volume, 200 μL; salt concentration, 10% w/v; vortex time, 2 min. Concentration of analytes were as follow: β-naphthol; 2.5, naphthalene; 2.5 and anthracene 0.125 μg mL^{-1}.

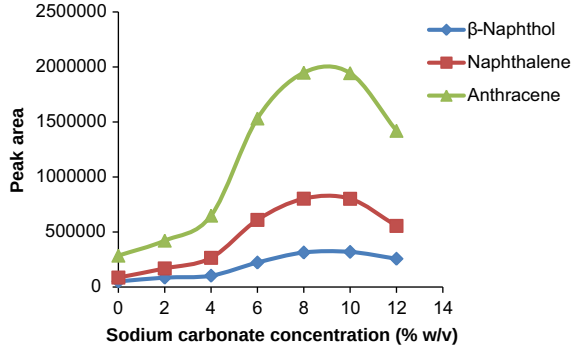

Fig. 6. Effect of ionic strength on the analytes extraction. Extraction conditions: extraction solvent, octanol/cyclohexane (50:50 v/v); extraction solvent volume, 200 μL; salt concentration, 10% w/v; vortex time, 2 min; aeration time; 5 min. Concentration of analytes were as follow: β-naphthol; 2.5, naphthalene; 2.5 and anthracene 0.125 μg mL^{-1}.

Table 1
Analytical features of VAALLME method.

Compound	Linear range (ng mL^{-1})	Slope	r^2	LOD (ng mL^{-1})	LOQ (ng mL^{-1})	Aver. EF[a]
β-Naphthol	50–2000	414,713	0.9949	10	50	29
Naphthalene	20–2000	833,654	0.9947	5.0	20	40
Anthracene	2–1000	2×10^6	0.9959	0.5	2.0	62

[a] Average enrichment factor in three concentration levels.

Table 2
Intraday and interday precision of VAALLME method.

Compound	Concentration (ng mL^{-1})	RSD (%)
Intraday (n=3)		
β-Naphthol	100	3.3
	500	3.1
	1000	3.4
Naphthalene	100	2.9
	500	2.6
	1000	3.4
Anthracene	10	2.1
	100	2.5
	500	2.3
Interday (n=9)		
β-Naphthol	100	3.5
	500	3.2
	1000	4.2
Naphthalene	100	5.1
	500	4.9
	1000	3.3
Anthracene	10	3.8
	100	4.3
	500	5.0

Table 3
Accuracy data for β-naphthol, naphthalene and anthracene spiked in real sample.

Compound	Concentration added (ng mL^{-1})	Concentration found (ng mL^{-1})	Recovery (%)	RSD % (n=3)
β-Naphthol	100	98.4	98.4	4.2
	500	495	99.0	3.4
	1000	1008	100.8	2.2
Naphthalene	100	97.8	97.8	4.4
	500	489	97.8	3.8
	1000	984	98.4	4.1
Anthracene	10	9.7	97.0	5.2
	100	102	102.0	4.5
	500	505	101.0	3.2

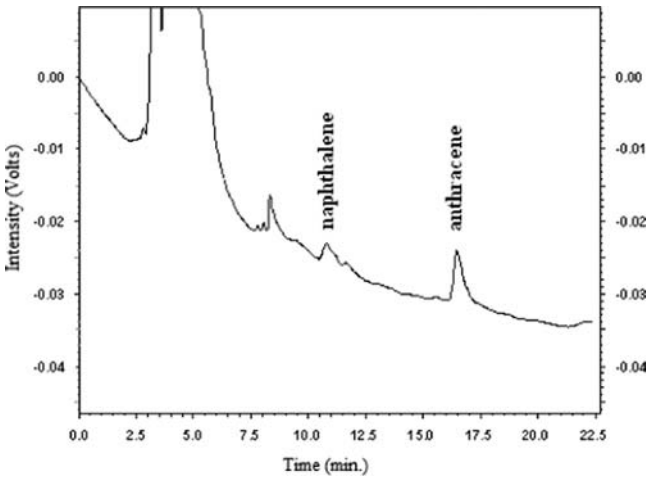

Fig. 7. HPLC chromatogram of real sample. Extraction conditions: extraction solvent, octanol/cyclohexane (50:50 v/v); extraction solvent volume, 200 μL; salt concentration, 10% w/v; vortex time, 2 min; aeration time; 5 min.

and then decreased. Finally, 10% w/v was chosen as a suitable concentration.

3.6. Effect of sample pH

The effect of sample pH on the extraction efficiency of analytes was studied in the range of 2 to 10. The results revealed that pH is not effective parameter in extraction of these analytes by using VAALLME method. The reason of this behavior can be attributed to the studied analytes, which do not participate in acid–base equilibriums. Consequently, the pH of sample solution was not adjusted for subsequent studies.

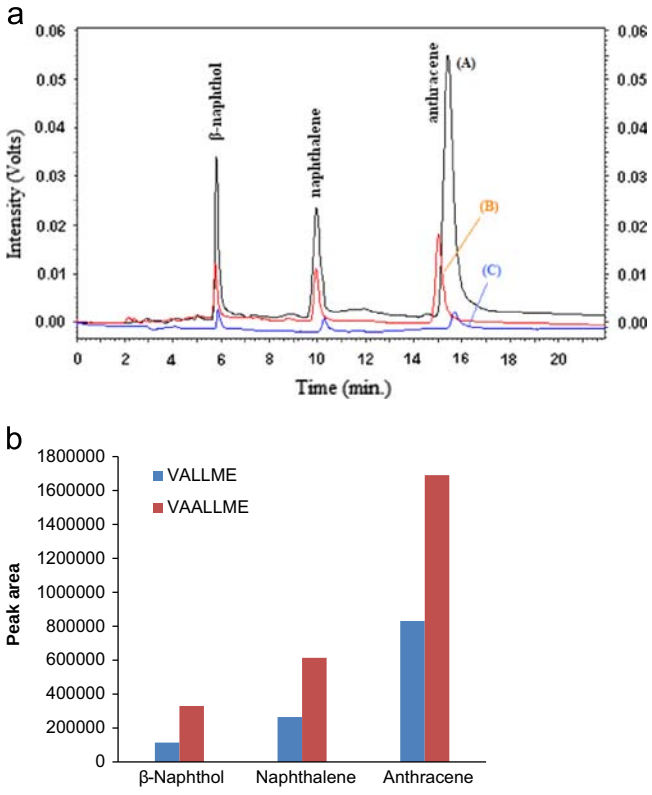

Fig. 8. Comparison of VALLME and VAALLME. (a) HPLC chromatograms of VAALLME (A), VALLME (B) and direct injection of standard solution (C). Concentration of analytes were as follow: β-naphthol; 2.5, naphthalene; 2.5 and anthracene 0.125 μg mL^{-1}. (b) Effect of two methods on analytes extraction.

3.7. Method evaluation

Linearity is the ability of the test method to provide results that are directly proportional to analyte concentration within a given range. The linearity of the proposed method was determined using standard solutions treated with the proposed method (VAALLME) under optimized conditions. The calibration curves for analytes over the desired concentration ranges exhibited good linearity. Results were shown in Table 1.

The limit of detection (LOD) was defined by the lowest detectable concentration yielding an $S/N=3$. On the other hand, limit of quantification (LOQ) was defined as concentration with $S/N=10$. LOD and LOQ values were presented in Table 1.

The repeatability of the VAALLME method was evaluated via analysis of three replicate experiments by spiking deionized water with three analytes at known concentrations. To study the intra-day and interday precision spiked samples were extracted by using the optimized proposed method in one and three different days in triplicate. Results were shown in Table 2. The relative standard deviations (RSDs) were below 5.0%.

In order to investigate the presence of matrix effects on the VAALLME method, a recovery study was carried out. Average recoveries and RSDs of the method were evaluated using spiked samples containing different concentrations of the analytes. The results in Table 3 indicate the VAALLME method gives acceptable recoveries for studied analytes.

3.8. Application of the proposed method to real sample

To evaluate performance of the proposed method, extraction and determination of β-naphthol, naphthalene and anthracene in wastewater sample were carried out under the optimized conditions that mentioned above. Figs. 7 and 8 show the typical chromatograms of wastewater sample, standard solution and deionized water spiked with the target analytes. Naphthalene and anthracene were found in the concentrations of 8.08 and 1.03 μg mL^{-1} in wastewater, respectively. β-Naphthol cannot be detected in this sample, indicating the sample was free of β-naphthol or probably the amount of this analyte was below the LOD of current method.

3.9. Comparison of the proposed method with VALLME method

To study the effect of aeration process, extraction of the analytes from a same aqueous sample solution was performed under optimized conditions by VALLME and VAALLME methods. In the VALLME technique, instead of the aeration step, samples were centrifuged. The results are shown in Fig. 8. The amounts of

Table 4
Comparison between analytical parameters of the proposed method and others methods in literature.

Analyte	Method	Matrix	LOD (ng mL^{-1})	LOQ (ng mL^{-1})	RSD (%)	Recovery (%)	Analysis time (min)[a]	Ref.
Anthracene	FA-HLLME-GC-FID[b]	Soil	24	40	8.07	90.0–111.0	≈30	[24]
Naphthalene	SPNE-GC–MS[c]	Water	0.514	1.7	3.1	82.6–87.6	≈80	[25]
Anthracene			0.624	2.06	4.2	91.2–95.6		
β-Naphthol	Derivitization-LLE-GC–MS	Fish and shellfish	10	–	< 5.04	86.0–91.0	≈150	[26]
Naphthalene	SA-SPE-LC-UV[d]	Sewage sludge	40	101	4	100	≈60	[27]
Anthracene			50	123	1	60		
β-Naphthol	VAALLME-HPLC-UV	Water and wastewater	10	50	< 4.2	98.0–100.8	≈30	PM[e]
Naphthalene			5	20	< 5.1	97.0–98.4		
Anthracene			0.5	2	< 5.0	97.0–102.0		

[a] Analysis time include sample preparation and chromatographic run time.
[b] Flotation-assisted homogeneous liquid–liquid microextraction (FA-HLLME) gas chromatography–flame ionization detector.
[c] Solid-phase nanoextraction (SPNE) gas chromatography–mass spectrometery.
[d] Sonication assisted-solid phase extraction (SA-SPE).
[e] Proposed method.

extracted analytes by using VAALLME method significantly are higher than VALLME.

The analytical parameters of the proposed method were compared with several reported methods in the literatures (Table 4). The results show the LODs and LOQs of target analytes were improved by using the VAALLME–HPLC–UV. In the other hand, analysis time of the VAALLME method was shorter than other methods. The proposed method can be surely used to determine of these analytes in water and wastewater samples in short time.

4. Conclusion

In this study for the first time, vortex and air- assisted liquid–liquid microextraction as a new sample preparation method was introduced and optimized using β-naphthol, naphthalene and anthracene as model analytes. The proposed method is based on a binary solvent system including aqueous sample and extraction solvent (octanol). Unlike the DLLME method, no disperser solvent and centrifuging were employed in the VAALLME procedure. Organic phase can be easily separated from aqueous phase by using aeration, which contributed to analytes extraction and increase the extraction efficiency. Extraction process can be simplified via automation.

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