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D-µ-SPE for Simultaneous Determination of Tetracycline and Bisphenol A in Water Samples using Liquid Chromatography

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The present study focuses on the application of dispersive micro solid-phase extraction (D-µ-SPE) in the quantitative analysis of tetracycline (TC) and bisphenol A (BPA) in water samples using high-performance liquid chromatography-diode array detector (HPLC-DAD) with graphene oxide (GO) as the sorbent. By systematically optimizing key parameters such as the amount of sorbent, extraction time, and sample volume, optimal conditions to achieve maximum extraction efficiency were obtained. They were determined to be 30 mg of sorbent, 15 min extraction time, and 15 mL sample volume. The method achieved excellent linearity, with the coefficient of determination (R^2) of 0.9985 for TC and 0.9789 for BPA. Depending on the analyte, the LOD and LOQ ranged from 2.41 - 2.57 µg L^{-1} and 6.7 - 8.6 µg L^{-1}. Subsequently, the developed method was successfully applied in the analysis of tap water samples, yielding relative recoveries ranging from 80.2 - 97.5% for TC and 86.2 - 100.5% for BPA, highlighting this method has its potential as a rapid, selective, and straightforward extraction method most suitable for routine water sampling analysis.

Keywords: Bisphenol A, tetracycline; dispersive micro solid-phase extraction; high-performance liquid chromatography; water samples

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Pharmaceuticals and Personal Care Products (PPCPs) are a group of emerging contaminants and have attracted increasing attention due to their adverse effects on human health and their potential bioaccumulation [1]. PPCPs' contaminants present in drinking water have generated significant concerns regarding the risk to humans. The large-scale use of pharmaceuticals has also increased their presence in surface water, groundwater, wastewater, and other water sources [2].

The environmental presence of PPCPs like tetracycline (TC) and Bisphenol A (BPA) has recently been a concern due to their potential to cause ecological harm and human health issues. TC contamination can contribute to developing antibiotic-resistant bacteria, a significant public health threat [3]. On the other hand, BPA can leach into water sources, affecting aquatic life and disrupting hormonal functions in both wildlife and humans. The environmental impact of BPA and tetracycline is profound. BPA can leach into water sources, affecting aquatic life by disrupting reproductive systems and development. Similarly, tetracycline residues can contribute to the development of antibiotic-resistant bacteria, posing a significant threat to environmental and public health [4].

Tetracyclines (TC) is an antibiotic that is mainly used as PPCPs. Tetracycline, on the other hand, is a broad-spectrum antibiotic used extensively in human and veterinary medicine and agricultural practices to promote growth and prevent disease in livestock [5]. The frequent use of tetracycline has resulted in significant amounts being released into the environment through improper disposal and agricultural and wastewater effluents. This has led to tetracycline residues in water bodies, soil, and food products. Wang and co-workers investigated water sources along the lower Yangtze River in China. In their report, 28 drinking water sources over dry, normal, and flood seasons were used to detect the amount of tetracyclines in the water samples [6].

Bisphenol A (BPA) is a synthetic compound that has been extensively used in various plastics and resins, commonly applied in the production of water bottles, food containers, and other consumer goods. Its widespread application has led to its pervasive presence in water bodies, soil, and even the atmosphere. The basic chemical structure of the BPA molecule involves two phenols together linked by different functional groups [7]. There are various sources of BPA, including air, water, food, PPCPs, and plastics. It is a high-volume production of organic synthetic compounds used to synthesize plastics [8]. It was initially used in the manufacturing of epoxy resins and polycarbonates. The current global production

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of BPA is about 8 million tonnes per year. Epoxy resins are extensively used in coil coatings, food and drink coatings, flooring, and adhesives [9]. Table 1 lists the physicochemical properties of both TC [10] and BPA [11].

Sample preparation is the most critical and laborious stage in analytical studies, and it may significantly affect the analytical information obtained in a particular chemical analysis [12]. The selected sample preparation method should be compatible with the subsequent steps in the analysis (i.e., dissolution, clean-up, fractionation, pre-concentration, or derivatization) [13]. These factors are strongly interdependent with one another, and failure to select a sample preparation method that is most compatible with these factors may result in experimental difficulty, loss of sample, prolonged analysis time, and high experimental cost [14].

Several advances exist in sample preparation for analyzing TC and BPA in various samples. Solid Phase Extraction (SPE) is the standard method for both TC and BPA. However, SPE has several drawbacks, such as tedious multiple steps, time consumption, and high solvent consumption. Recently, dispersive micro solid phase extraction (D-µ-SPE) has been utilized in the analysis of target analytes and has shown many advantages, such as very little solvent consumption, effective cleanup, and shorter procedure [15]. Moreover, it is simple and rapid, produces good recovery, and can be combined with different detection techniques [16].

In this study, a comprehensive optimization and method development using dispersive micro solid phase extraction (D-µ-SPE) assisted with Liquid Chromatography to simultaneously determine two compounds of TC and BPA in water samples was employed. Three parameters, namely, the amount of sorbent, the extraction time, and the sample volume, were investigated using one factor at a time (OFAT) method. OFAT was used as the optimization method at the initial screening phase. Graphene oxide (GO) was utilized as the sorbent in D-µ-SPE extraction for the pre-concentration in TC and BPA in water samples.

It is important to investigate the amount of TC and BPA in water samples due to their risk to human health. The method used in this work could provide additional advantages to the simultaneous application of TC and BPA analysis. This method is expected to be selective, sensitive, rapid, consume less organic solvent, reduce cost, and be efficient.

EXPERIMENTAL

Chemicals and Materials

Graphene oxide (GO) was purchased from GO Advance Solutions in Serdang, Malaysia. All the reagents are analytical grade. Standard tetracycline (TC) was obtained from Fisher Chemicals (Loughborough, UK). Bisphenol A (BPA) was obtained from Sigma-Aldrich (USA). Methanol (MeOH), acetonitrile (ACN), and ethanol (EtOH) of high-performance liquid chromatography (HPLC grade) were purchased from Merck (Darmstadt, Germany). The deionized water was prepared with an SZ-2 system from Shanghai Lu West Analytical Instruments (Shanghai, China).

Preparation of Standard and Stock Solutions

At a concentration of 1000 mg L^{-1}, the individual stock solutions of BPA and TC were prepared separately in HPLC grade methanol, and all of it was stored in amber glass bottles at 4 °C. A series of standard working solutions were prepared in methanol using a dilution process. The tap water and river water samples were collected and cleaned with acetone. The pre-cleaned samples were filtered with a nylon membrane filter and stored in a freezer at 4 °C. Hydrochloric acid (HCl) or sodium hydroxide (NaOH) was used to modify the pH of the aqueous solution. The calibration curve was created using the integrated peak areas versus concentrations of compounds.

Table 1. Physico-chemical properties of Tetracyclines (TC) and Bisphenol A (BPA).

Name	Tetracycline (TC)	Bisphenol A (BPA)
Molecular Formula	$C_{22}H_{24}N_2O_8$	$C_{15}H_{16}O_2$
Molecular Weight (g/mol)	444.4	228.3
Melting point	172.5℃ - 223℃	158℃ -159℃
Solubility (at 25℃)	231 mg L^{-1}	120 mg L^{-1}
logKow	-1.37	3.4
pH	3.0 - 7.0	2.0 - 2.5
pKa (at 25℃)	3.30	9.8 - 10.4

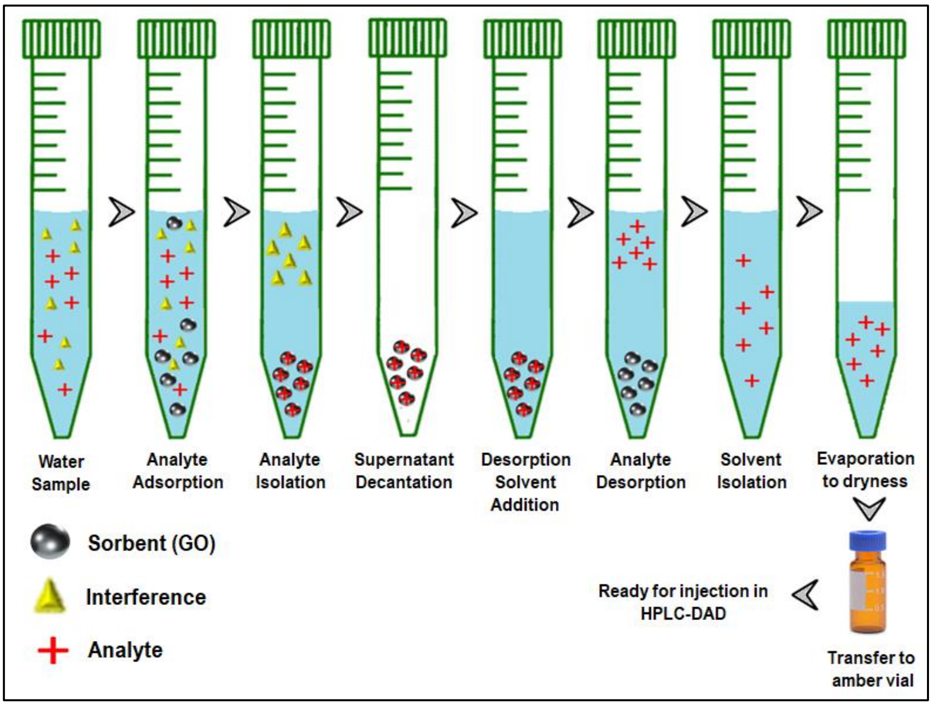

Figure 1. Schematic diagram of dispersive micro solid phase extraction (D-µ-SPE) procedure using GO as the sorbent.

Chromatographic Conditions

The determination of the desired analytes was conducted using an HPLC system (Agilent G1313A HPLC) equipped with a diode array detector (DAD) RPC18 column with a 20 µL sample loop. The analytical reversed-phase C18 column (Agilent G1313A HPLC) equipped with a diode array detector (DAD), RPC18 column particle diameter, 4.6 mm internal diameter: 25 cm long and 20 µL sample loop was employed for the separation process. Synthesis of BPA and TC were chromatographically separated using the isocratic mobile phase of acetonitrile-water (85/15, v/v) at a flow rate of 1.0 mLmin^{-1}. The wavelength used was 270 nm.

Dispersive Micro Solid Phase Extraction (D-µ-SPE) Procedure

0.3 g of GO powder was taken and dispersed into 10 mL of sample solution with a pH of 3 in a 50 mL centrifuge tube. The mixture was stirred vigorously to trap the analytes using a magnetic stirrer for about 15 min. The analytes were then injected using a 1 mL syringe into the sample solution to allow for the absorption of the analytes into the dispersed sorbent. The GO powder was isolated by centrifugation from the solution at 5000 rpm for 3 min. For another 15 min, the desorption solvent was added into the centrifuge of 2 mL of grade methanol. The mixture was then centrifuged at 5000 rpm for 3 min. The collected solvent was evaporated under a nitrogen gas stream. 1 mL of extracted analyte was transferred into a 1 mL amber glass vial. Finally, 25 µL of the extract was injected into the HPLC system. Figure 1 below shows the schematic diagram of the dispersive micro solid phase extraction (D-µ-SPE) procedure using GO as the sorbent.

Optimization of Parameters

The parameters that needed to be analyzed were the mass of Graphene Oxide (GO) as sorbent, extraction time, and sample volume. The optimization process was carried out using one factor at a time (OFAT) while other parameters were kept constant. The results were then used to evaluate the method's extraction efficiency. Thus, the optimum conditions of D-µ-SPE were used in the real sample analysis.

Validation Methods

The validation of the analytical method in D-µ-SPE implemented in this research was to demonstrate that the analytical procedure was reliable and suitable for the desired purposes, including linearity, limit of detection (LOD), and limit of quantification (LOQ), precision, and accuracy.

Limit of Detection (LOD) and Limit of Quantification (LOQ)

The Limit of Detection (LOD) and Limit of Quantification (LOQ) were determined based on linear regression of the calibration curve. The formula of LOD and LOQ are as follows:

$$LOD = 3\sigma / m$$

$$LOQ = 10\sigma / m$$

Where

σ = standard deviation

m = slope

RESULTS AND DISCUSSION

Optimization of D-µ-SPE Parameters

In this study, the optimization of three D-µ-SPE extraction parameters of sample volume, extraction time, and mass of sorbent were investigated. The optimization process was carried out using OFAT.

Effect of Mass of Sorbent

The experiment was carried out by varying the mass of sorbent to 20, 30, 40, and 50 mg of GO powder. From Figure 2, it was found that with 30 mg of sorbent, the highest peak area of analytes was obtained, and with 50 mg sorbent, the lowest peak area was observed. However, there was no significant increase in the peak area when the mass of sorbent was increased. As the mass of sorbent increases, the peak continues to decrease. This could be due to the excessive mass of sorbent, which results in the difficulty of desorption, requiring a higher volume of desorption solvent and the capacity of sorbent to become saturated [17, 18]. Thus, 30 mg was chosen as the mass of sorbent for the subsequent experiments.

Effect of Extraction Time

Based on the previous experiment, the optimum mass of sorbent (30 mg) was employed in this section with 15 min extraction time. The optimization of extraction time was carried out by varying the time to 5, 15, and 30 min. As shown in Figure 3, the highest extraction time was 15 min after the extraction, and subsequently, the peak slowly decreased. This could be due to the long duration that causes back extraction of analytes from the acceptor into the sample solution [19, 20]. Thus, 15 min was chosen as the optimum extraction time and was used in subsequent analysis.

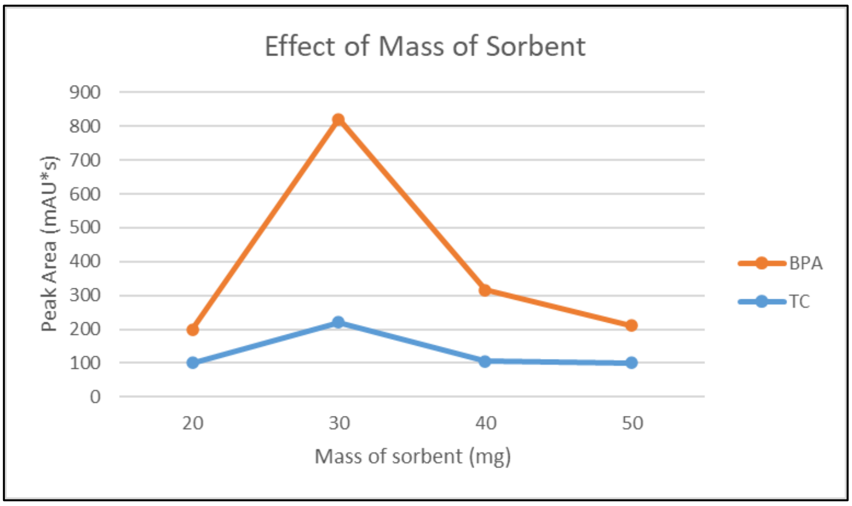

Figure 2. Mass of sorbent vs. peak area in the extraction of BPA and TC.

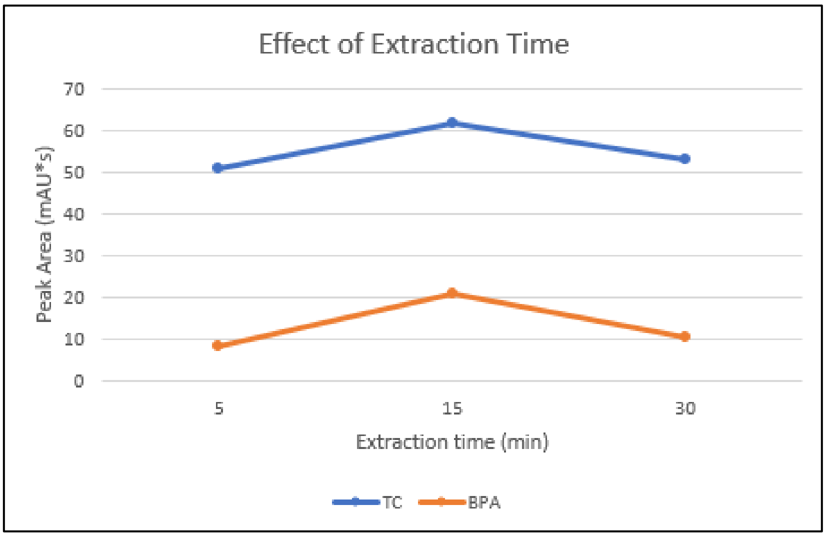

Figure 3. Graph showing extraction time vs peak area in the extraction of BPA and TC.

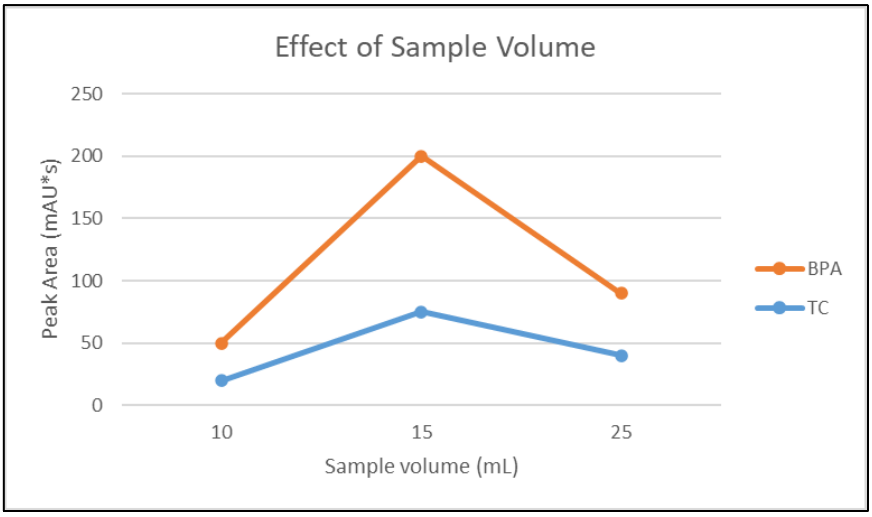

Figure 4. Sample volume vs peak area in the extraction of BPA and TC.

Effect of Sample Volume

The sample volume is an essential factor in determining the loading capacity of d-SPE extraction [21]. The mass was kept to 30 mg in optimizing the sample volume, and the extraction time was maintained at 15 min. Three different sample volumes (10 - 25 mL) were investigated. As shown in Figure 4, the highest peak area was observed with a volume of 15 mL, which indicates the optimum sample volume for high extraction efficiency. Nevertheless, a gradual decrease in extraction was observed at 25 mL of the samples.

Method Validation and Analytical Performance of D-µ-SPE

The optimum conditions for the D-µ-SPE method were validated for its relative recovery, linearity, and precision. Based on Table 2, good linearity from the range of 5-500 µg L^{-1} was obtained for the analytes where the coefficient of determination obtained were 0.9985 and 0.9789. The LOD values obtained were 2.41 µg L^{-1} for TC and 2.57 µg L^{-1} for BPA analyte. The LOQ values obtained were 6.671 µg L^{-1} for TC and 8.6 µg L^{-1} for BPA analyte. The precision was calculated by percent relative standard deviation (% RSD), and the results obtained were 3.2 % for TC and 4.7 % for BPA.

Table 2. Validation Data of D-µ-SPE of TC and BPA in Water Sample.

Sample	Analytes	Linear range (µg L^{-1})	Coefficient determination (R^2)	LOD (µg L^{-1})	LOQ (µg L^{-1})	Precision (RSD, %) (*n*=3)
Tap water	TC	5-500	0.9985	2.41	6.7	3.2
	BPA	5-500	0.9789	2.57	8.6	4.7

Table 3. Relative Recoveries (%) of D-µ-SPE from three different concentrations of spiked water sample.

Analyte	Spike Concentrations (µg L^{-1})	Relative Recovery (%)	RSD % (*n=3*)
TC	10	80.2	2.7
	100	83.3	1.85
	500	97.5	1.5
BPA	10	86.2	3.2
	100	90.1	0.8
	500	100.5	2.1

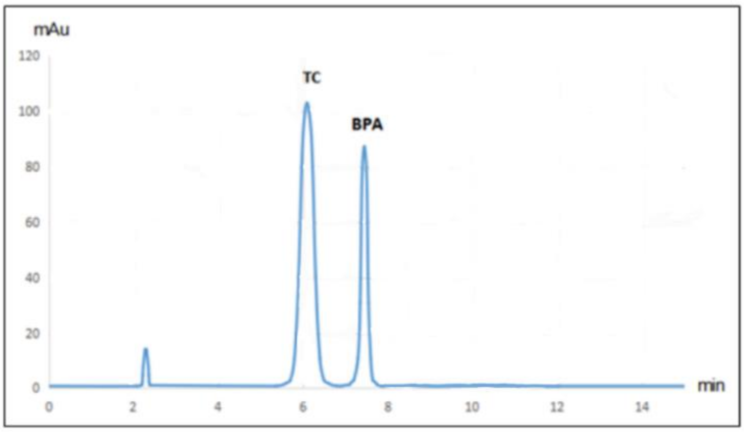

Figure 5. Chromatogram of mixed TC and BPA (100 µg L^{-1}) in Tap Water Sample.

Relative recovery was conducted by spiking tap water to give the three different final concentrations of 10 µg L^{-1}, 100 µg L^{-1}, and 500 µg L^{-1}. Results shown in Table 3 show good relative recoveries for both analytes in the range of 80.2% - 100.5% and excellent reproducibility with a relative standard deviation (RSD) of < 3.2%.

Figure 5 shows the chromatogram of spiked tap water at a concentration of 100 µg L^{-1} of mixed TC and BPA. The results show that both analytes have been successfully extracted and well separated from the tap water samples. The chromatogram also shows that the peak of BPA is broader than that of TC because interferences, such as a metal chelator in the TC, could affect the extraction process [18].

The performance of D-µ-SPE in the quantitative analysis of TC and BPA in water samples using HPLC-DAD with graphene oxide (GO) as the sorbent was compared with other reported methods regarding the sensitivity of method validation (Table 4). The developed method exhibited an excellent limit of detection and an acceptable relative recovery percentage comparable to the conventional SPE-HPLC technique.

Table 4. Comparison With Other Methods on the Sensitivity of Method Validation Using SPE for TC and BPA.

Analyte	Analysis Method	Type of sample	Linear range (µg L^{-1})	LOD (µg L^{-1})	Recoveries (%)	Ref.
TC	SPE-HPLC	Food sample (fish and chicken)	4-1000	1.2 to 2.7	Fish sample: 91.1-114.1 Chicken sample: 69.5-93.2	[22]
	µ-SPE-HPLC	honey	10–5000	15.3	89-94	[23]
	SPE-HPLC	Food Sample (Milk)	20–1000	10-20	82-104	[24]
	D-µ-SPE-HPLC	Tap water	5-500	2.41	80.2 - 97.5	This work
BPA	MSPE-HPLC	Environmental Sample (Wastewater)	1-200	0.5-0.8	77.60-89.60	[25]
	µ-SPE-HPLC	Food Sample (Canned fruits)	2 to 10	7.00	87.1-108.9	[26]
	SPE-HPLC	Food Sample (milk)	5–200	1.32	111.6	[27]
	D-µ-SPE-HPLC	Tap water	5-500	2.57	86.2 - 100.5	This work

MSPE-HPLC: magnetic solid-phase extraction high-performance liquid chromatography
SPE-HPLC: solid-phase extraction high-performance liquid chromatography
µ-SPE-HPLC: micro-solid-phase extraction high-performance liquid chromatography

CONCLUSION

Results from this study show that the dispersive micro solid phase extraction (D-µ-SPE) can be optimized using the OFAT method and successful analysis of analytes using High-Performance Liquid Chromatography (HPLC). 30 mg of mass sorbent, 15 min of extraction time, and 15 mL of sample volume were the parameters for the optimum conditions to achieve the highest peak area. Both analytes, TC and BPA, were successfully extracted using these optimum conditions in D-µ-SPE. Good linearities were achieved for the analytes, with the coefficient of determination (R^2) found to be 0.9985 (TC) and 0.9789 (BPA). The method was applied to analyze tap water samples with good relative recoveries of 80.2% - 100.5%. The D-µ-SPE method in the extraction of BPA and TC can be considered a selective, simple, fast, efficient, and green extraction method that could be used in the laboratory for water sample analysis.

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REFERENCES

1. Li, G., Deng, R., Peng, G., Yang, C., He, Q., Lu, Y. & Shi, H. (2018) Magnetic solid-phase extraction for the analysis of bisphenol A, naproxen and triclosan in wastewater samples. *Water Science and Technology*, **77(9)**, 2220–2227.

2. Ebele, A. J., Abou-Elwafa Abdallah, M. & Harrad, S. (2017) Pharmaceuticals and personal care products (PPCPs) in the freshwater aquatic environment. *Emerging Contaminants*, **3(1)**, 1–16.

3. Yi, L., Zuo, L., Wei, C., Fu, H., Qu, X., Zheng, S. & Zhu, D. (2020) Enhanced adsorption of bisphenol A, tylosin, and tetracycline from aqueous solution to nitrogen-doped multiwall carbon nanotubes via cation-π and π-π electron-donor-acceptor (EDA) interactions. *Science of the Total Environment*, **719**, 137389.

4. Ben, Y., Fu, C., Hu, M., Liu, L., Wong, M. H. & Zheng, C. (2019) Human health risk assessment of antibiotic resistance associated with antibiotic residues in the environment: A review. *Environmental research*, **169**, 483–493.

5. Daghrir, R. & Drogui, P. (2013) Tetracycline antibiotics in the environment: a review. *Environmental chemistry letters*, **11**, 209–227.

6. Wang, Z., Chen, Q., Zhang, J., Dong, J., Yan, H., Chen, C. & Feng, R. (2019) Characterization and source identification of tetracycline antibiotics in the drinking water sources of the lower Yangtze River. *Journal of Environmental Management*, **244**, 13–22.

7. Vasiljevic, T. & Harner, T. (2021) Bisphenol A and its analogues in outdoor and indoor air: Properties, sources and global levels. *Science of the Total Environment*, **789**, 148013

8. Othman, N. Z., Hanapi, N. S. M., Ibrahim, W. N. W. & Saleh, S. H. (2020) Alginate Incorporated Multi-Walled Carbon Nanotubes as Dispersive Micro Solid Phase Extraction Sorbent for Selective and Efficient Separation of Acidic Drugs in Water Samples. *Nature Environment and Pollution Technology*, **19(3)**, 1155–1162.

9. Tess, R. W. (2018) Epoxy resin coatings. In *Epoxy Resins*, *Routledge*, 719–782.

10. Daghrir, R. & Drogui, P. (2013) Tetracycline antibiotics in the environment: a review. *Environmental Chemistry Letters*, **11**, 209-227.

11. Juan, D. A., Hasan, H. A., Muhamad, M. H., Abdullah, S. R. S., Abu Bakar, S. N. H. & Buhari, J. (2021) Physico-chemical and biological techniques of bisphenol A removal in an aqueous solution. *Journal of Ecological Engineering*, **22(9)**, 136–148.

12. Mitra, S. (Ed.) (2004) *Sample preparation techniques in analytical chemistry*, *John Wiley & Sons*.

13. Pedersen-Bjergaard, S., Gjelstad, A. & Halvorsen, T. G. (2015) Sample preparation. *Bioanalysis of Pharmaceuticals: Sample Preparation, Separation Techniques, and Mass Spectrometry*, 73–122.

14. Lingeman, H. & Tjaden, U. R. (2021) Sample pretreatment procedures In *Detection-oriented derivatization techniques in liquid chromatography*, *CRC Press*, 85–151.

15. Zaini, N., Hanapi, N. S. M., Ibrahim, W. N. W., Osman, R., Kamaruzaman, S., Yahaya, N. & Anis, A. L. (2022) Dispersive Micro-Solid Phase Extarction (D-µ-SPE) with Polypyrrole-Grapheen Oxide (PPY-GO_ Nanocomposite sorbent for the Determination of Tetracycline Antibiotics in Water. *Malaysian Journal of Analytical Sciences*, **26(5)**, 953–964.

16. Chisvert, A., Cárdenas, S. & Lucena, R. (2019) Dispersive micro-solid phase extraction. *TrAC Trends in Analytical Chemistry*, **112**, 226–233.

17. Ruiz, F. J., Ripoll, L., Hidalgo, M. & Canals, A. (2019) Dispersive micro solid-phase extraction (DµSPE) with graphene oxide as adsorbent for sensitive elemental analysis of aqueous samples by laser induced breakdown spectroscopy (LIBS). *Talanta*, **191**, 162–170.

18. Ghorbani, M., Aghamohammadhassan, M., Ghorbani, H. & Zabihi, A. (2020) Trends in sorbent development for dispersive micro-solid phase extraction. *Microchemical Journal*, **158**, 105250.

19. Abdullah, U. A. A. U., Hanapi, N. S. M., Ibrahim, W. N. W., Hadzir, N. M., Zaini, N., Anis, A. L. & Yahaya, N. (2021) Pre-Concentration and Determination of Tetracyclines Antibiotics Residues in Water Samples Using RGO/Fe 3 O 4 Nanocomposite as Extraction Sorbent. *Indonesian Journal of Chemistry*, **21(5)**, 1196–1211.

20. Chung, W. H., Tzing, S. H. & Ding, W. H. (2013) Dispersive micro solid-phase extraction for the rapid analysis of synthetic polycyclic musks using thermal desorption gas chromatography–mass spectrometry. *Journal of Chromatography A*, **1307**, 34–40.

21. Lawal, A., Wong, R. C. S., Tan, G. H., Abdulra'uf, L. B. & Alsharif, A. M. A. (2018) Multi-pesticide residues determination in samples of fruits and vegetables using chemometrics approach to QuEChERS-dSPE coupled with ionic liquid-based DLLME and LC–MS/MS. *Chromatographia*, **81**, 759–768.

22. Li, P., Rao, D., Wang, Y. & Hu, X. (2022) Adsorption characteristics of polythiophene for tetracyclines and determination of tetracyclines in fish and chicken manure by solid phase extraction-HPLC method. *Microchemical Journal*, **173**, 106935.

23. Arabsorkhi, B. & Sereshti, H. (2018) Determination of tetracycline and cefotaxime residues in honey by micro-solid phase extraction based on electrospun nanofibers coupled with HPLC. *Microchemical Journal*, **140**, 241–247.

24. Wu, X. J., Wang, G. N., Yang, K., Liu, H. Z. & Wang, J. P. (2017) Determination of tetracyclines in milk by graphene-based solid-phase extraction and high-performance liquid chromatography. *Analytical Letters*, **50(4)**, 641–650.

25. Li, G., Deng, R., Peng, G., Yang, C., He, Q., Lu, Y. & Shi, H. (2018) Magnetic solid-phase extraction for the analysis of bisphenol A, naproxen and triclosan in wastewater samples. *Water Science and Technology*, **77(9)**, 2220–2227.

26. Abdullah, U. A. A. U., Hanapi, N. S. M., Ibrahim, W. N. W., Saim, N. A. & Yahaya, N. (2018) Micro-solid phase extraction (µ-SPE) based on alginate/multi-walled carbon nanotubes sorbent for the determination of bisphenol A in canned fruits. *Chiang Mai J. Sci.*, **45(6)**, 2348–2360.

27. O'Mahony, J., Moloney, M., McCormack, M., Nicholls, I. A., Mizaikoff, B. & Danaher, M. (2013) Design and implementation of an imprinted material for the extraction of the endocrine disruptor bisphenol A from milk. *Journal of Chromatography B*, **931**, 164–169.