# Determination of Selected Organic Cosmetic Ingredients in Brackish Water Samples

Edyta Kudlek, Ewa Łobos-Moysa, and Ewa Felis

Abstract—The identification of a bride range of organic pollutants in real water samples, especially in brackish water or wastewater is still a major issue in chemical analysis. The paper presents a comparison of different solid phase extraction SPE methods for the preparation of brackish water samples before the chromatographic analysis. The SPE methods were dedicated to the extraction of selected organic cosmetic ingredients - butylated hydroxytoluene BHT, oxybenzone BP3, and octyl methoxycinnamate OMC. Six types of SPE cartridges with different bed types and weights were tested. The SPE methods included the conditioning of the column bed with organic solvents, loading of the pre-treated brackish water samples, washing out the impurities, and the elution of the analytes. The highest recovery of the tested cosmetic ingredients, which has reached the value of 100% for BP3 and OMC, and exceeded 99% for BHT, was noted for the cartridge with the C<sub>18</sub> (octadecyl) bed.

*Index Terms*—Brackish water, organic micropollutants, SPE, gas chromatography.

# I. INTRODUCTION

The production and use of personal care products are considered an important environmental risk [1], [2]. Personal care products include in their composition different types of pharmaceutically active substances, disinfection agents, UV filters, synthetic antioxidants, parabens, phthalates, glycols, preservatives, and others. Those chemicals can be released into the aquatic environment and harm living organisms. High concentrations of those compounds occur not only in wastewater from factories producing cosmetics but also can be noted in swimming pool water, marine water, domestic wastewater, and brackish effluents from landfills [3]-[6]. Those highly polluted water matrixes cause many problems during their analysis, especially during chromatographic analyses. The organic analytes which occur mainly in low concentrations should be properly isolated from inorganic compounds and organic contaminants of high molecular weight.

Solid phase extraction SPE belongs to the major routinely utilized method for extracting different types of micropollutants from a bride range of water matrixes [7], [8]. SPE was commercialized in the late 1970s and replaced the

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liquid-liquid extraction [9]. The use of this type of extraction method is in accordance with EPA guidelines for aqueous sample analysis [10]. The SPE technique involved the use of small amounts of sorbents, called column bed, placed between two filter layers in a cartridge. They are many types of column beds, which differ from each other due to the used sorbent, its amount, and composition [11].

The column bed should be activated before it came to contact with the water sample. The activation is usually performed by the use of different organic solvents and water of a given pH. The analysed water samples often required a pre-treatment procedure which allows for the separation of undesirable undissolved ingredients which can block the SPE cartridge during the extraction process. The samples could be also in this step spiked by an internal standard (IS), which improves the reliability of results especially in samples in which we expect a large matrix effect [12]. The pre-treatment of the sample increase also the sorption of the analytes on the column bed and affects the recovery value. The undesirable compounds which were adsorbed on the column bed after the extraction of the sample could be removed by an additional washing step of the SPE cartridge before the elution of analytes. The elution of analytes in the greatest number of cases takes place by the use of the same solvent which was used for the column bed activation. The obtained extract could be directly subjected to chromatographic analysis or could be additionally concentrated, to increase the concentration of analytes.

The development of universal methods for sample preparation is a very difficult and actual topic in chemical analysis. Particular attention should be paid to the analysis of personal care product ingredients which relatively non-polar and are more soluble and better extracted in relatively nonpolar organic solvents [13]. The bride range of compounds belonging to this group makes it difficult to apply one strictly defined analytical method and often requires an individual approach to each compound.

The conducted study aimed to develop a procedure for determining selected cosmetic ingredients occurring in brackish water samples. The paper presents a selection of water sample preparation procedures based on SPE conducted on different types of extraction cartridges. The most favorable extraction procedure was chosen based on the recovery of tested compounds and the LOQ value calculated by the use of gas chromatography results.

#### II. MATERIALS AND METHOD

The brackish water was collected from an enterprise located in the Silesian Voivodeship in Poland from a storage

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reservoir. The characteristic of brackish wastewater was presented in table I. The brackish water samples were spiked with three organic cosmetic ingredients - butylated hydroxytoluene BHT, oxybenzone BP3, and octyl methoxycinnamate OMC. The concentration of the individual organic compound was set at 0.5, 1.0, 2.0, 5.0, and 10.0 ng  $\mu$ L<sup>-1</sup>. The tested brackish water before the insertion of the chosen cosmetic ingredients types did not contain this type of substance in its composition. The analytical standards of BHT, BP3, and OMC were supplied by Merck KGaA (Darmstadt, Germany). Table II summarized the chemical structures and the general properties of the tested compounds.

TABLE I: CHARACTERISTIC OF BRACKISH WATER

| Parameter                                       | Value |
|---|-------|
| total organic carbon (TOC), mgC L <sup>-1</sup> | 764   |
| рН, -   | 8.3   |
| conductivity, mS cm <sup>-1</sup>               | 12.1  |
| salinity, mg NaCl L <sup>-1</sup>               | 7500  |
| chloride, mgCl <sup>-</sup> L <sup>-1</sup>     | 1600  |
| inorganic carbon (IC), mgC L <sup>-1</sup>      | 1231  |
| colour, mgPt/L                                  | 2600  |

TABLE II: CHARACTERISTICS OF TESTED ORGANIC COSMETIC INGREDIENTS

| Standard | Structural formula  | Molecular<br>formula | Molar<br>mass,<br>g mol <sup>-1</sup> | CAS<br>number |
|----------|---|----------------------|---------------------------------------|---------------|
| BHT      | CH <sub>3</sub> OH CH <sub>3</sub><br>H <sub>3</sub> C CH <sub>3</sub><br>CH <sub>3</sub> CH <sub>3</sub> | $C_{15}H_{24}O$      | 220.35                                | 128-37<br>-0  |
| BP3      | O OH<br>OCH3  | $C_{14}H_{12}O_3$    | 228.24                                | 131-57<br>-7  |
| OMC      | H <sub>3</sub> CO   | $C_{18}H_{26}O_3$    | 290.40                                | 5466-7<br>7-3 |

Organic solvents such as acetonitrile and methanol used for the conditioning of the SPE cartridges and the extraction were of purity grade >99.5% and >99.8% respectively and were purchased from Avantor Performance Materials Poland S.A. (Gliwice, Poland). The SPE was conducted on six types of disposable cartridges from Supelco Inc (Bellefonte, USA), which were compared in table III. The extracts collected after SPE were analyzed using a gas chromatograph coupled to mass spectrometry GC-MS(EI), Model 7890B by Perlan Technologies (Warszawa, Poland). 1.0 µL of each extract was separated in a SLB<sup>TM</sup>-5 ms Capillary GC Column of Supelco Inc (Bellefonte, USA) with an internal diameter of 0.25 mm, a length of 30 m, and a layer thickness of 0.25  $\mu$ m. The operating parameters of the GC-MS(EI) were selected based on previous research devoted to the analysis of organic micropollutants in matrices with a lower concentration of organic and inorganic compounds [14], [15]. The GC oven temperature program was as follows: 80 °C (6 min), 5 °C/min to 260 °C, 20 °C/min to 300 °C. Helium with a flow of 1.1 mL/min was used as the support phase. The injector work in the splitless mode and its temperature was set at 250  $\,$ °C. The temperature of the ion source and trap were 230 °C and 150 °C respectively. The ion recording mode works in the range from 50 to 650 m/s. The precision of the quantitative analysis was improved by the method of internal standards (IS-mirex).

| TABLE III: CHARACTERISTICS OF THE TESTED SPE CARTRIDGES |          |                         |                     |                       |
|---|----------|-------------------------|---------------------|-----------------------|
| Symbol  | Bed type | Carbon<br>loading,<br>% | Bed<br>weight,<br>g | Tube<br>volume,<br>mL |

| Symbol  | Bed type               | loading, | weight, | volume, |
|---------|------------------------|----------|---------|---------|
|         |                        | %        | g       | mL      |
| ENVI-8  | C <sub>8</sub> (octyl) | 14.0     | 1.0     | 6.0     |
| LC-8    | C <sub>8</sub> (octyl) | 7.0      | 0.5     | 6.0     |
| ENVI-18 | C18 (octadecyl)        | 17.0     | 1.0     | 6.0     |
| LC-18   | C18 (octadecyl)        | 11.5     | 1.0     | 6.0     |
| LC-CN   | Cyano                  | 7.0      | 0.5     | 6.0     |
| LC-Ph   | Phenyl                 | 5.5      | 0.5     | 3.0     |

#### III. MATH RESULTS AND DISCUSSION

The optimum experimental conditions for the extraction and quantification of all chosen cosmetic ingredients from a brackish water waste stream were investigated employing an experimental design procedure, which includes the check of the linearity of the mass detector response, the pre-treatment of the brackish water before the SPE and the choice of the most favorable SPE column and solvent composition.

# A. The Linearity Check of the Mass Detector Response

The calibration of the mass detector was conducted based on calibration curves prepared for each tested organic compound. The standard solutions of BHT, BP3, and OMC were prepared in methanol in a concentration range from 0.5 to 10 ng/ $\mu$ L. Five injections of all tested compound standard solutions were made. The linearity of the mass detector response was checked by the use of linear regression. Table IV summarized the retention time t<sub>R</sub> of the cosmetic ingredient in the chosen temperature program of the GC oven. The standard deviation SD of the estimated retention time did not exceed 0.02 min. Retention times of the tested organic compounds were significantly different from each other. These allow for proper separation and appropriate identification of those compounds complex water matrices such as wastewater and brackish water [16].

The correlation coefficient  $R^2$ , which was not less than 0.95, indicated good linearity of the detector's response for both, BHT, BP3, and OMC.

TABLE IV: RETENTION TIME  $T_{R}$  of the Cosmetic Ingredient in the Chosen Temperature Program of the GC Oven

| Cosmetic ingredient | Retention time,<br>t <sub>R</sub> min | Standard deviation<br>of t <sub>R</sub> , SD min | Correlation coefficient, R <sup>2</sup> |
|---------------------|---------------------------------------|--|---|
| BHT                 | 15.49                                 | 0.01   | 0.95                                    |
| BP3                 | 22.46                                 | 0.02   | 0.99                                    |
| OMC                 | 24.01                                 | 0.01   | 0.98                                    |

#### B. The Repeatability of Quantitative Results

The identification of organic impurities in different types of water matrixes and the assessment of their concentration by a chosen detection technique should be repeatable [17]. The repeatability was expressed by the coefficient of variation (CV) and presented for five concentrations of the tested cosmetic ingredient in table V. The obtained CV values did not exceed 2.31%, confirming acceptable repeatability of the conducted measurement technique.

TABLE V: THE COEFFICIENT OF VARIATION FOR DIFFERENT CONCENTRATION LEVELS OF COSMETIC INGREDIENT

| CONCENTRATION LEVELS OF COSMETIC INGREDIENT |                            |                          |                          |                          |                           |         |
|---|----------------------------|--------------------------|--------------------------|--------------------------|---------------------------|---------|
| Cosmetic                                    | С                          | oefficient               | of variati               | on, CV %                 | )                         | LOD, ng |
| ingredient                                  | 0.5 ng<br>μL <sup>-1</sup> | 1 ng<br>μL <sup>-1</sup> | 2 ng<br>μL <sup>-1</sup> | 5 ng<br>μL <sup>-1</sup> | 10 ng<br>μL <sup>-1</sup> | L-1     |
| BHT   | 1.93                       | 1.87                     | 1.06                     | 0.98                     | 0.87                      | 0.05    |
| BP3   | 1.32                       | 1.41                     | 2.28                     | 2.08                     | 0.95                      | 0.02    |
| OMC   | 2.22                       | 2.31                     | 2.14                     | 1.74                     | 1.55                      | 0.01    |

The limit of detection (LOD), which was also given in table V, ranged from 0.05 ng L-1 for BHT to 0.01 ng L-1 for OMC. The values of LOD give an overview of the lowest amount of an organic compound that can be distinguished from the absence of that compound keeping a certain confidence interval [18].

# C. Sample Pre-treatment and Preparation Procedure

The proper preparation of the sample before the main analysis is the key factor of environmental sample analysis. The preparation step should allow for the isolation of chosen analytes, which may occur in the given sample, taking into account a reduced solvent consumption, the improvement of the extraction throughout, higher compound recoveries, and a better reproducibility [19]. As mentioned before the SPE is one of the most commonly used extraction techniques for a bride range of organic micropollutants including cosmetic ingredients. The extraction of analytes from samples with a complex organic and inorganic composition forces the implementation of a pre-treatment step. Chen et al. [20] pointed that the filtration of samples can prevent obstruction of the SPE cartridges by suspended substances, such as suspended particles, colloids, and microorganisms that can occur in the sample. In our case, the standards of the tested cosmetic ingredients were introduced in the brackish water in a mixture with the concentration level of each particular compound of 1 mg  $L^{-1}$ . The prepared solution was filtered through a glass microfiber filter with a pore size of 0.45 µm followed by a filter from the same material with a pore size of 0.20 µm. The pH of the obtained filtrate was adjusted to 7.0 by 0.1 M HCl and subjected to the tested SPE cartridges. The steps of the SPE procedure were illustrated in figure 1.

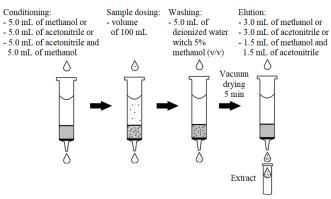


Fig. 1. SPE procedure steps.

The selection of the most preferable extraction conditions was performed by searching for the appropriate combination of SPE cartridge type and organic solvents used for the cartridge bed conditioning and the elution of the analytes. Recovery and limit of quantification (LOQ) of the chemicals were examined for each method of sample preparation. The obtained results were listed in Table VI. Based on these two parameters, the most optimal methodology for examined cosmetic ingredients extraction was chosen.

Conditioning with 5 mL of acetonitrile followed by 5 mL of methanol and extraction of the analytes by the use of the ENVI-18 tube was considered the best suited for BP3 and OMC. In the case of BHT, the highest recovery and the lowest LOQ value was noted during the use of the ENVI-8 cartridge, the bed of which was conditioned with methanol and after the sample load and cleaning eluted also by single methanol. The obtained results correspond to those noted during the method development for the analysis of compounds belonging to the group of contaminants of emerging concern occurring in swimming pool water [14] or deionized water samples [15]. The worst recovery results of the tested cosmetic ingredients were noted for the LC-CN cartridge. Also, the LOQ values achieved after the use of the LC-Ph cartridges did not meet the expectations of a correct, accurate, and sensitive method of sample analysis.

TABLE VI: RETENTION TIME  $\ensuremath{\mathsf{TR}}$  of the Cosmetic Ingredient in the Chosen Temperature Program of the GC Oven

| Cosmetic   | etic SPE |                         |          |              | g and elution             |
|------------|----------|-------------------------|----------|--------------|---------------------------|
| ingredient |          | Parameter               | Methanol | Acetonitrile | Methanol and acetonitrile |
| BHT        | ENVI-8   | Recovery, %             | 100      | 98.5         | 98.6                      |
|            |          | LOQ, ng L <sup>-1</sup> | 0.13     | 0.83         | 0.68                      |
|            | LC-8     | Recovery, %             | 100      | 95.6         | 97.2                      |
|            |          | LOQ, ng L <sup>-1</sup> | 0.22     | 0.58         | 0.42                      |
|            | ENVI-18  | Recovery, %             | 98.9     | 97.4         | 98.7                      |
|            |          | LOQ, ng L <sup>-1</sup> | 0.55     | 1.97         | 1.69                      |
|            | LC-18    | Recovery, %             | 96.2     | 93.8         | 96.2                      |
|            |          | LOQ, ng L <sup>-1</sup> | 1.06     | 1.39         | 1.99                      |
|            | LC-CN    | Recovery, %             | 75.6     | 73.9         | 81.6                      |
|            |          | LOQ, ng L <sup>-1</sup> | 2.11     | 3.45         | 2.64                      |
|            | LC-Ph    | Recovery, %             | 79.3     | 70.4         | 78.3                      |
|            |          | LOQ, ng L <sup>-1</sup> | 2.57     | 3.59         | 3.87                      |
| BP3        | ENVI-8   | Recovery, %             | 98.5     | 96.7         | 99.5                      |
|            |          | LOQ, ng L <sup>-1</sup> | 3.07     | 2.45         | 3.88                      |
|            | LC-8     | Recovery, %             | 81.7     | 95.5         | 98.4                      |
|            |          | LOQ, ng L <sup>-1</sup> | 2.97     | 7.22         | 1.18                      |
|            | ENVI-18  | Recovery, %             | 100      | 89.7         | 100                       |
|            |          | LOQ, ng L <sup>-1</sup> | 2.11     | 3.17         | 0.95                      |
|            | LC-18    | Recovery, %             | 70.2     | 76.1         | 100                       |
|            |          | LOQ, ng L <sup>-1</sup> | 4.55     | 8.34         | 2.94                      |
|            | LC-CN    | Recovery, %             | 99.7     | 98.6         | 85.7                      |
|            |          | LOQ, ng L <sup>-1</sup> | 3.56     | 2.12         | 8.56                      |
|            | LC-Ph    | Recovery, %             | 99.8     | 69.2         | 89.3                      |
|            |          | LOQ, ng L <sup>-1</sup> | 2.87     | 5.05         | 7.15                      |
| OMC        | ENVI-8   | Recovery, %             | 84.9     | 82.3         | 86.5                      |
|            |          | LOQ, ng L <sup>-1</sup> | 1.06     | 0.99         | 0.98                      |
|            | LC-8     | Recovery, %             | 82.7     | 81.5         | 83.1                      |
|            |          | LOQ, ng L <sup>-1</sup> | 1.15     | 1.08         | 1.02                      |
|            | ENVI-18  | Recovery, %             | 99.8     | 99.9         | 100                       |
|            |          | LOQ, ng L <sup>-1</sup> | 0.06     | 0.09         | 0.05                      |
|            | LC-18    | Recovery, %             | 99.9     | 98.3         | 100                       |
|            |          | LOQ, ng L <sup>-1</sup> | 0.18     | 0.16         | 0.14                      |
|            | LC-CN    | Recovery, %             | 69.6     | 79.6         | 75.9                      |

|       | LOQ, ng L <sup>-1</sup> | 1.55 | 1.45 | 1.36 |
|-------|-------------------------|------|------|------|
| LC-Ph | Recovery, %             | 71.3 | 65.4 | 62.1 |
|       | LOQ, ng L <sup>-1</sup> | 1.98 | 2.02 | 1.87 |

Table VII summarized the recovery and LOQ for lower concentrations of the chosen cosmetic ingredients by the implementation of the above-mentioned sample pre-treatment procedure and the SPE conditions chosen according to the most favourable cartridge type and solvent combination:

- Analysis of BHT ENVI-8 cartridge conditioned with methanol, elution by 3 mL of methanol;
- Analysis of BP3 and OMC ENVI-18 cartridge conditioned with acetonitrile and methanol, elution by 1.5 mL of methanol and 1.5 mL of acetonitrile.

TABLE VII: RECOVERY FOR DIFFERENT CONCENTRATIONS OF COSMETIC INGREDIENTS DURING OPTIMAL EXTRACTION CONDITIONS

| Concentration of compounds in brackish water, $mg L^{-1}$ | Cosmetic ingredient | Recovery $\pm$ SD, % |
|---|---------------------|----------------------|
| 0.5   | BHT                 | $100\pm1.2$          |
|   | BP3                 | $100\pm0.4$          |
|   | OMC                 | $100\pm0.5$          |
| 0.2   | BHT                 | $100\pm1.8$          |
|   | BP3                 | $99.7\pm1.4$         |
|   | OMC                 | $98.3\pm2.1$         |
| 0.1   | BHT                 | $98.2\pm3.6$         |
|   | BP3                 | $97.7\pm4.1$         |
|   | OMC                 | $95.5\pm5.9$         |

The obtained results indicated that the recovery of compounds decreased with de decrease in their concentration in the tested water sample. However, the recovery values for compound concentrations equal to 1.0 mg L<sup>-1</sup> were equal to those noted for the 0.5 mg L<sup>-1</sup> compound concentration. The calculated recovery factors qualified the accuracy of the results obtained from the chosen analytical method as very good. This was also confirmed by the repeatability of the results SD, which value was in the range from 0.4 to 5.9%.

### IV. CONCLUSION

The conducted research allowed for the development of a methodology for the trace determination of three chosen cosmetic ingredients - BHT, BP3, and OMC in brackish water. It was proven that the presented analytical procedure based on the pre-treatment of water samples and their solid phase extraction by the use of ENVI-8 and ENVI-18 cartridges enables the quantification of micropollutants with satisfactory accuracy and repeatability. The developed method could be also successfully applied for monitoring of different types of cosmetic ingredients and other organic micropollutants which occur not only in brackish but also in other types of highly polluted water. It should be noted that the physicochemical composition of water affects the qualitative and quantitative analysis of water samples and forces the development of sample preparation methods that not only allow for the proper identification of compounds but also protect the analytical equipment. Extended analysis of the qualitative and quantitative composition of water samples allows the selection of appropriate technologies for their treatment. An accurate and repeatable method guarantees a proper assessment of the effectiveness of the proposed technologies and contributes to the monitoring of the risk of harmful effects of pollutants on the natural environment.

#### CONFLICT OF INTEREST

The authors declare no conflict of interest.

# AUTHOR CONTRIBUTIONS

Edyta Kudlek and Ewa Łobos-Moysa conceived and designed the experiments. Edyta Kudlek performed the experiments, and analyzed the data together with Ewa Łobos-Moysa and Ewa Felis. Ewa Łobos-Moysa contributed reagents, materials, and analysis tools. Edyta Kudlek wrote the paper. All authors had approved the final version.

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