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# Determination of synthetic musks in the sediment of the Taihu lake by using accelerated solvent extraction (ASE) and GC/MS

Jin-Shui Che<sup>ab</sup>, Rui-Peng Yu<sup>b</sup>, Qi-Jun Song<sup>a\*</sup>, Li-Ping Wang<sup>b</sup> and Sheng-Fang Wu<sup>b</sup>

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Synthetic musks, substitutes for natural musks, are widely distributed in environment. They have been detected in water, sludge, fish, shrimp, mussels and other aquatic animals, and even in human's adipose tissue, blood and breast milk. In this study, a new extraction procedure, based on the accelerated solvent extraction (ASE) and in cell clean-up technique was developed and successfully coupled with gas chromatography-mass spectrometry (GC/MS) for the analysis of musks in sediment samples. With this method, the limits of detection as low as  $0.03-0.05 \text{ ng g}^{-1}$  and the recovery rate of 86.0%-104% are achieved. When compared with soxhlet extraction (SE) and ultrasonic extraction (USE), ASE not only has the best extraction efficiency but also has advantage in extraction time and solvent consumption. Eight synthetic musks, including six polycyclic musks (Tonalide (AHTN), Galaxolide (HHCB), Phantolide (AHDI), Traseolide (ATII), Cashmeran (DPMI) and Celestolide (ADBI)) and two nitro musks (musk xylene (MX) and musk ketone (MK)), were evaluated in sediment samples collected from 15 selected locations of the Taihu lake, one of the largest freshwater lakes in China. The contents of synthetic musks in sediment samples range from 0.336 to  $3.10 \text{ ng g}^{-1}$  for HHCB, 0.184 to  $1.21 \text{ ng g}^{-1}$  for AHTN, below detection limit (BDL) to  $0.349 \text{ ng g}^{-1}$  for MX, and BDL to  $0.0786 \text{ ng g}^{-1}$  for MK. The contents of DPMI, ADBI, AHMI and ATII are below detection limit in all samples. The results reflect current status of fragrance compound pollution in this area, and provide basic data for environmental policy making.

Keywords: synthetic musks; sediment; accelerated solvent extraction (ASE); Taihu lake

# 1. Introduction

Since the discovery of the first synthetic musk compound by Baur in 1888, several kinds of musks have been synthesised around the 1950s [1,2]. As cheap substitutes for natural musks, synthetic musks are widely used in many personal care products, such as perfume, creams, body lotion, shampoo, soap, and laundry soap powder etc [3,4].

Synthetic musks can be divided into three major subgroups: nitro musks (e.g. MX, MK), polycyclic musks (e.g. AHTN, HHCB, AHDI, ATII, DPMI, ADBI) and macrocylic

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musks (e.g.Muscone and Exaltone) [2]. Currently, polycyclic musks dominate the synthetic musks usage in the fragrance industry [2,5]. The structures of some synthetic musks are shown in Figure 1 [2].

After production and use, synthetic musks may enter the sewage system and be distributed widely in the aquatic environment. In 1981, the occurrence of nitro musk (MX) in biological samples and river water was confirmed for the first time by Japanese scientists, and polycyclic musks were detected for the first time in 1994 [4]. In the following decade, synthetic musks were widely detected in water, sludge, fish, shrimp, mussels and other aquatic animals, and even in people's adipose tissue, blood and breast milk [2,4].

Recently, synthetic musks have raised considerable attention in the field of environmental chemistry. Synthetic musks were confirmed to be difficult to degrade, and they are easily bioaccumlated through the food chain [5]. The toxicities of synthetic musks have been extensively studied. For example, it was reported that the nitromusks are effective inhibitors of multixenobiotic resistance transporters in marine mussels. As such they may enhance the toxic potential of other multixenobiotic resistance substrates by enabling them to accumulate in the cells [6]. Nitromusks (MX and MK) are carcinogenic to mice, and they can cause the variation of mouse enzyme cytochrome P450 (CYP) [7–9]. There is some evidence indicating that the metabolites (aniline transformation formation products) may be more hazardous than the parent compounds of nitromusks [2,10]. As a result of these concerns, nitromusks have been banned, restricted, or listed as chemicals of concern in several countries [2,11]. Polycyclic musks have been shown to be both estrogenic and anti-estrogenic in many bioassays (both in vitro and in vivo) [12]. The inhibition of the multi-xenobiotic defenses of mussel gill cells by HHCB and AHTN was also reported [12,13]. At relatively high dietary exposure levels, an increase in mice liver weight was evidenced [14-16].



Figure 1. The chemical structures of some synthetic musks.

Various analytical approaches for the extraction and determination of synthetic musks in environmental and biological samples were proposed in recent years. The extraction of musks from sediment was often carried out with Soxhlet extraction (SE) [9,12]. Ultrasonic extraction (USE), due to its simplicity in operation and inexpensive instrument, was also used. However, these techniques are time-consuming and require large volumes of organic solvents [20]. In the last few years, a new extraction technique, accelerated solvent extraction (ASE), has been developed to reduce the volume of extraction solvents, extraction time and to improve the recoveries of analytes. ASE, also known as pressurised liquid extraction (PLE), represents a highly effective extraction technique [15,17], which takes advantage of the increased analyte solubility at temperatures well above the boiling points of common solvents. The kinetic processes involved for analytes to desorb from the matrix are accelerated at high pressure, and the whole extractions process can be automated [17–19]. The ASE is widely applicable for environment and food analysis to replace some of the above-mentioned solvent- and time-consuming techniques [20–22]. For example, the ASE method has been used in extracting the musks in sediment, sludge and biological tissues. After the ASE extraction, however, the solid phase extraction (SPE) or gel permeation chromatography (GPC) methods are often needed for the clean-up procedures [25-28], leading to prolonged sample preparation time. In this study, a "in cell clean-up" procedure for the purification process was employed to solve the time-consuming problem.

China is one of the biggest producers and exporters of the synthetic musks [1]. In China, however the impact of synthetic musks on the environment has not been fully aware, and the information relating to the synthetic musks in the environment is very limited for the public as well as for policy makers. Thus, the aim of this study is to compare the different extraction methods and to establish a fast and convenient method for synthetic musks determination in sediment samples.

#### 2. Experimental

# 2.1 Chemical and materials

Synthetic musks (DPMI, ADBI, AHMI, ATII, AHTN, HHCB, MX, MK) and two internal standards, hexamethyl benzene (HMB), AHTN-D3, were purchased from Dr. Ehrenstorfer GmbH, Augsburg, Germany. Florisil (60–100 mesh, Sigma-Aldrich Laborchemikalien GmbH, USA), was activated at 450°C for 5 h before extraction. Other reagents used in this work were obtained from Sinopharm Chenical Reagent Co., Ltd, China. Analytical grade *n*-hexane and acetone were distilled before use. Anhydrous sodium sulfate was dried at 450°C for 5 h before it was used in the sample preparation procedure.

# 2.2 Sampling

The Taihu Lake, located between Jiangsu and Zhejiang Province, is the third largest freshwater lake in China. The total area of Taihu Lake is about  $36500 \text{ km}^2$ , it serves a population of 34,000,000. All of the sediment samples were collected from 14-15 June in 2009. The specific locations of the sampling points are shown in Figure 2. After removal of large solid particles, the sediments were homogenised and stored in PTFE bottles. Upon returning to the laboratory, the samples were frozen at  $-20^{\circ}$ C, and freeze-dried



Figure 2. Map of the sampling points. 1. Yuan-tou-zhu; 2. Sha-tang port; 3. Jiao-shan; 4. Da-pukou; 5. Ping-tai-shan; 6. Da-lei-shan; 7. Xin-tang port; 8. No.4 signal lamp; 9. Xiao-mei-kou; 10. Xin-gang-kou; 11. Pu-zhuang; 12. Xu-kou; 13. Yu-yang-shan; 14. Man-shan; 15. Wu-gui-shan.

(LABCONCO-Freezone stoppering Tray dryer, USA) as soon as possible. These samples were ground to pass through 100-mesh sieve, and kept in the dark and in a dry place before use.

# 2.3 Extraction procedures

# 2.3.1 ASE extraction

Sediment samples were extracted by ASE using Dionex ASE 100 equipped with an 11 mL stainless steel extraction cell. For each sample, one or two filter membranes (Dionex Corporation) were put at the bottom of the ASE extraction cell, which can play the role of filtering and prevent the tiny powder blocking the extraction cell. An amount of 2 g Florisil and 5 g freeze-dried sediment powder were transferred subsequently into the sample cell, and then internal standards HMB and AHTN-D3 were added. Florisil was used for in cell clean-up, to adsorb a small quantity of impurity in the sediment samples. The sample was extracted with two 5 min cycles at a pressure of 1500 psi and temperature of 100°C. The other instrument parameters were set as follow: flush volume 60%, flushing time 90s, preheating time 3min. The extract was concentrated to near dry with rotary evaporator and subsequently with nitrogen blow. After re-dissolving the residue with 1 mL hexane, the sample solution was subjected to GC/MS analysis.

# 2.3.2 Soxhlet extraction (SE)

The standard-spiked samples were packaged in filter paper and placed in Soxhlet extraction thimble. Extraction was carried out with 200 mL n-hexane/acetone (1:1, v/v)

for 8 h in a classical Soxhlet extractor. After concentration, the extract was re-dissolved in 20 mL ultra-pure water and subjected to SPE purification [6]. The HC-C18 SPE cartridges (500 mg, 3 mL) were activated subsequently by 5 mL methanol and 5 mL ultra-pure water. The extracts pass through the cartridges at a flow rate of  $2 \text{ mL min}^{-1}$ . A volume of 5 mL n-hexane was used to elute the analytes at a flow rate of  $1 \text{ mL min}^{-1}$ . The eluate was reduced to a final volume of 1 mL using nitrogen blow before GC/MS analysis.

# 2.3.3 Ultrasonic extraction (USE)

The standard-spiked samples were added into a 50 mL beaker, and extracted with 20 mL n-hexane/acetone (1:1, V/V) for 10 min in an ultrasonic bath (40 W). The extraction was repeated two additional times and the supernatants were combined. The combined supernatants were filtered, and the filtrate was used for the subsequent processing. The concentration and the clean-up procedures that follow the extraction were the same as that described in Section 2.3.2.

# 2.4 Instrumental analysis

All the analysis was carried out with a GC/MS instrument. The gas chromatography (VARIAN CP3800) was equipped with an Auto-sampler (Combi PAL GC Multi-Function, CTC Analytics, Swiss). A fused-silica capillary column DB-WAX (30 m, 0.25 mm i.d.×0.25 µm film thickness, Agilent Technologies, USA) was used for the separation. The injection was performed in a splitless mode with a volume of 1 µL and the injector temperature of 270°C. Helium was used as the carrier gas with a flow rate of 1 mL min<sup>-1</sup>. The column oven temperature was programmed from 120°C (hold for 0.5 min) to 230°C (hold for 8.5 min) at a rate of 10°C min<sup>-1</sup>. The mass spectrometry detector (VARIAN 1200L Quadrupole MS/MS) was operated in selected ion monitoring (SIM). The capillary column was coupled directly to the ion source, which was operated in electron impact (EI) ionization mode. The transfer line, ion source temperatures and emission current were 250°C and 200°C, and 50 µA, respectively.

### 2.5 Data quantification

Data quantification and processing were carried out with a VARIAN Workstation System (Version 6.9). The analytes were identified and verified by comparing the retention time and mass spectra of sample with the corresponding standard. Data acquisition was performed using the following characteristic ions: DPMI(191, 206), ADBI(229, 244), AHMI(229, 244), HHCB(243, 213), ATII(215, 258), AHTN(243, 258), MX(292, 297), MK(279, 294), and two internal standard HMB(147, 162), AHTN-d3(246, 243) [12]. The selected ion chromatograms, extracted from the total ion current (TIC), were integrated for the analyte quantification. The chromatograms obtained from the standards and a sediment sample are exemplified in Figures 3 and 4, respectively.

#### 2.6 Quality control

Considering the wide application of musks in household products, great care was taken to avoid contamination that may occur during sample preparation procedures.



Figure 3. SIM Chromatograms of synthetic musks standards. All the concentrations of the musk standards were  $20 \text{ ng mL}^{-1}$ .



Figure 4. SIM Chromatograms of a sediment sample.

Blanks experiments were carried out to ensure that no musk was detectable in the water and reagents used in this experiment.

Recovery experiments were performed with the procedure described in Section 2.3.1. The musk standards (DPMI, ADBI, AHMI, HHCB, ATII, AHTN, MX and MK) were added to the sediments, and then they were subjected to the same analytical procedure as for the samples. The recovery rates of DPMI, ADBI, AHMI, HHCB, ATII, AHTN, MX, MK were 81.3%, 90.4%, 88.9%, 104%, 81.8%, 91.7%, 93.2%, 86.0%, respectively. The relative standard deviations (RSD) were in the range of 0.5 to 6.9%, indicating an acceptable repeatability for all analytes.



Figure 5. The recovery rates obtained with different solvents. The standard deviations of the data are represented with error bars, n = 3.

The limits of detection (LODs) and limits of quantification (LOQs) for each analyte were obtained on the basis of a signal-to-noise ratio of 3 and 10, respectively [12,22]. The LODs of DPMI, ADBI, AHMI, HHCB, ATII, AHTN, MX, MK were 0.050, 0.050, 0.050, 0.030, 0.050, 0.050, 0.050 ng  $g^{-1}$ , respectively. The LOQs were 0.16, 0.16, 0.16, 0.10, 0.16, 0.16 ng  $g^{-1}$  for DPMI, ADBI, AHMI, HHCB, ATII, AHTN, MX, MK, respectively. Only peaks exceeded LOQ were quantified.

Concentrations of each musk were obtained by the external standard calibration method based on a 4-point calibration curve. Calibration curves for all investigated musks showed a  $R^2$  value better than 0.99, namely 0.997, 0.998, 0.998, 0.996, 0.996, 0.996, 0.995, 0.995 for DPMI, ADBI, AHMI, HHCB, ATII, AHTN, MX, MK, respectively.

#### 3. Results and discussion

# 3.1 Optimisation of ASE

#### 3.1.1 Optimisation of solvent

The selection of solvent is dependant not only on the analyte, but also on the kind of adsorbent used in an extraction procedure, as solvent may affect the adsorptivity of an adsorbent [29] (see Section 3.2). Many solvents were proposed for the extraction of synthetic musks, including *n*-hexane, *n*-hexanedichlormethane (DCM), *n*-hexane/acetic ester, *n*-hexane/acetone [2]. In order to obtain the best performance, the extraction efficiencies of these solvents were compared. The best recovery was obtained when a mixture of *n*-hexane and acetone (1:1, V:V) was used and the recoveries for the four analysed musks were in the range of 86.0–104% (Figure 5). The recoveries obtained with *n*-hexane, *n*-hexane/dichlormethane (DCM) (1:3, V:V), and *n*-hexane/acetic ester (1:1, V:V) were in the range of 72.0–83.3%, 68.9–87.6%, and 81.3–102%, respectively. Therefore, a mixture of *n*-hexane and acetone (1:1, V:V) was chosen as the extraction



Figure 6. The effect of extraction temperature on the recovery rates. The standard deviations of the data are represented with error bars, n = 3.

solvent in this work, the selection was found to be coincident with the report for the polycyclic aromatic hydrocarbons (PAHs) extraction from soil [23].

# 3.1.2 Optimisation of extraction temperature

Temperature is one of the most important parameters that affect the ASE efficiency, elevated temperature can increase desorption kinetics of target compounds, and also lower viscosity of the solvent. Consequently, the analytes could diffuse into the solvent matrix more readily [11,18]. On the other hand, the higher temperature, the more impurity may be extracted [29]. In this study, we tested four different temperatures for the extraction experiment, i.e.,  $60^{\circ}$ C,  $80^{\circ}$ C,  $100^{\circ}$ C and  $120^{\circ}$ C. The highest recovery rates were obtained with the extraction temperature at  $100^{\circ}$ C for all the musks, they were in the range of 86.0-104% (Figure 6). This extraction temperature was in accordance with that of Ternes's study, where  $100^{\circ}$ C was also selected as the extraction temperature [22]. In  $120^{\circ}$ C, the recovery rates were decreased, which may be explained by the relatively low boiling points of synthetic musks ( $110^{\circ}$ C– $130^{\circ}$ C) [2]. When the temperature reaches  $120^{\circ}$ C, the standards of synthetic musks may evaporate from the extraction cell.

# 3.1.3 Optimisation of extraction cycle

The extraction efficiency of the ASE technique depends on the number of extraction cycles. In this study, we selected 5 min as the static extraction time, since this duration was reported to be appropriate in similar works [22]. In the first extraction cycle, the recovery rates of HHCB, AHTN, MX and MK were, respectively, 80.6%, 77.0%, 77.1% and 71.3%, these figures were increased to 95.7%, 91.7%, 93.2% and 86.4%, respectively in the second extraction cycle. After the third extraction cycle, these figures were increased to 96.5%, 93.6%, 93.9% and 88.4%, respectively (Figure 7). As only marginal increase in the recovery rates was observed, it was not necessary to conduct the third extraction cycle. From



Figure 7. The recovery rates obtained with different extraction cycles.

the data presented in the Figure 7, it can be seen that two extraction cycles with 5 min of static extraction were adequate for the overall recoveries.

#### 3.2 Purification process

After the extraction, it is critical to remove the impurities that may be co-extracted from the samples; therefore a purification process is often needed. The contents of impurities are determined by the solvent, the temperature and the adsorbent used in the extraction procedure [29]. In many previous studies, the purification process was conducted by solid phase extraction (SPE). For example in Ternes's study [22], SPE was used as a purification method following the ASE extraction, however, the recovery rates were only about 64% and 74% for HHCB and AHTN, respectively.

In this paper, we chose a method of "in cell clean-up" with Florisil, as described in the technical notes TN210 of the Dionex Corporation [29]. With this method, a very satisfactory result was obtained, the recovery rates were markedly higher than that obtained by the SPE method. Furthermore, the impurity peaks were small and exhibited no interference to the analysis. The in cell clean-up can also reduce the experiment steps and simplify the overall experimental procedure.

# 3.3 Comparison with other extraction methods

In this study, the overall percentage recoveries obtained with the different extraction techniques (SE, USE and ASE) were compared (also see Section 2.3). The results are presented in Figure 8. It was noted that the best recovery rates were obtained from the method of ASE, where the recovery rates were 104%, 91.7%, 93.2% and 86.0% for HHCB, AHTN, MX and MK, respectively. The recovery rates obtained with the Soxhlet extraction were 95.4%, 92.0%, 85.0% and 80.3% respectively for HHCB, AHTN, MX and MK, they were slightly lower than that obtained by the ASE method. The recovery



Figure 8. The recovery rates obtained with different extraction methods. The standard deviations of the data are represented with error bars, n = 3.

rates of ultrasonic extraction (USE) for the four musks were around 75%, which were significantly lower than the recovery rates of the ASE.

The overall experiment time (including extraction time, purification time, and instrumental analysis time) for ASE, USE and SE were about 1 h, 2 h and 10 h, respectively. The overall solvent cost for ASE, USE and SE were estimated to be 20 mL, 50 mL and 200 mL, respectively. We may conclude, therefore, that in addition to the advantage of high recovery rates for all the musks, ASE was also solvent-saving and time-saving. Furthermore, the whole extraction procedure of ASE was automated with no need for an additional purification process.

### 3.4 Synthetic musks in the sediment samples

In eight synthetic musks, HHCB, AHTN, MX and MK were detected in the sediment samples of the Taihu Lake, whereas the DPMI, ADBI, AHMI, ATII were below detection limits (Table 1). According to the results, the HHCB, AHTN were detected in all the samples, the content of HHCB was from 0.336 to  $3.10 \text{ ng g}^{-1}$ , and AHTN was from 0.184 to  $1.21 \text{ ng g}^{-1}$ . The two nitro musks, MX and MK, were detected in most of the samples, MX from BDL to  $0.349 \text{ ng g}^{-1}$ , MK from BDL to  $0.0786 \text{ ng g}^{-1}$ . In terms of sampling points, relatively high concentrations of synthetic musks were found in position 1, 7, 9, 11, i.e., those locations near the edge of the lake (Figure 2). Close to position 1, 7, 9, there were several entrances to the lake, which may be accountable for the high concentration of synthetic musks. Most places with the low concentrations of synthetic musks were located far away from the edge or entrance to the lake.

Zeng *et al.* [30] and Zhang *et al.* [31] reported the concentrations of synthetic musks of Zhujiang River and Suzhou Creek in China. The concentration of levels of HHCB, AHTN were 3-121,  $7-167 \text{ ng g}^{-1}$  for Zhujiang River, and 3-78,  $2-31 \text{ ng g}^{-1}$  for Suzhou Creek, respectively, and the DPMI, ADBI, ATII, MX, MK were below the detection

| Samples |                  | DPMI | ADBI | AHMI | ННСВ  | AHTN  | ATII | MX    | MK     |
|---------|------------------|------|------|------|-------|-------|------|-------|--------|
| 1.      | Yuan-tou-zhu     | BDL  | BDL  | BDL  | 3.10  | 1.04  | BDL  | BDL   | BDL    |
| 2.      | Sha-tang port    | BDL  | BDL  | BDL  | 0.156 | 0.872 | BDL  | 0.151 | 0.0768 |
| 3.      | Jiao-shan        | BDL  | BDL  | BDL  | 0.474 | 0.263 | BDL  | 0.106 | BDL    |
| 4.      | Da-pu-kou        | BDL  | BDL  | BDL  | 0.815 | 0.326 | BDL  | 0.169 | BDL    |
| 5.      | Ping-tai-shan    | BDL  | BDL  | BDL  | 0.992 | 0.689 | BDL  | 0.112 | BDL    |
| 6.      | Da-lei-shan      | BDL  | BDL  | BDL  | 0.348 | 0.274 | BDL  | 0.100 | 0.0514 |
| 7.      | Xin-tang-port    | BDL  | BDL  | BDL  | 1.21  | 0.227 | BDL  | BDL   | 0.0449 |
| 8.      | NO.4 signal lamp | BDL  | BDL  | BDL  | 0.773 | 0.522 | BDL  | 0.349 | 0.0663 |
| 9.      | Xiao-mei-kou     | BDL  | BDL  | BDL  | 1.06  | 0.439 | BDL  | 0.168 | BDL    |
| 10.     | Xin-gang-kou     | BDL  | BDL  | BDL  | 0.403 | 0.308 | BDL  | BDL   | 0.0700 |
| 11.     | Pu-zhuang        | BDL  | BDL  | BDL  | 1.86  | 1.21  | BDL  | BDL   | BDL    |
| 12.     | Xu-kou           | BDL  | BDL  | BDL  | 0.336 | 0.211 | BDL  | BDL   | BDL    |
| 13.     | Yu-yang-shan     | BDL  | BDL  | BDL  | 0.409 | 0.436 | BDL  | BDL   | 0.0638 |
| 14.     | Man-shan         | BDL  | BDL  | BDL  | 0.652 | 0.184 | BDL  | 0.108 | BDL    |
| 15.     | Wu-gui-shan      | BDL  | BDL  | BDL  | 0.375 | 0.369 | BDL  | BDL   | BDL    |
|         |                  |      |      |      |       |       |      |       |        |

Table 1. Content of synthetic musk in the sediment samples of the Taihu lake  $(ngg^{-1} dry weight)$ .

BDL: below detection limit.

limits in all samples. The concentrations of musks in the Taihu Lake were lower than that of Zhujiang River and Suzhou Creek. The relative low concentration may result from the reduced discharge of wastewater into the lake. Since the outbreak of algae in the Taihu Lake in 2007 [33], the local governments paid more attention to the wastewater discharge in the surrounding area. In the case of Zhujiang River, the high concentration of musks may be explained by its location. The river flows through the Guangdong Province, one of the biggest provinces in China that produce the daily-used chemicals, which are the main source of the synthetic musks. For the Suzhou Creek, the surrounding area is more densely populated when compared to the area of Taihu Lake.

Aaron *et al.* [24] reported the concentrations of synthetic musks in Lake Erie and Lake Ontario of the USA and Canada, the concentrations of HHCB was  $3.2 \text{ ng g}^{-1}$  for Lake Erie, and the concentrations of HHCB, AHTN, ATII, AHMI, ADBI, MX in Lake Ontario were  $16 \text{ ng g}^{-1}$ ,  $0.96 \text{ ng g}^{-1}$ ,  $0.27 \text{ ng g}^{-1}$ ,  $0.049 \text{ ng g}^{-1}$ ,  $0.1 \text{ ng g}^{-1}$ ,  $0.068 \text{ ng g}^{-1}$ , respectively. The concentrations of nitromusks in San Francisco Bay were reported by Rubinfeld *et al.* [11], they were  $0.13-0.24 \text{ ng g}^{-1}$  for MX and  $1.08-2.74 \text{ ng g}^{-1}$  for MK. The contents of synthetic musks in the sediment of the Taihu Lake were a similar level compared to the data reported in the USA. Although the contents of synthetic musks in the Taihu Lake were not too high in comparison with that reported in other areas, considering the vast territory and huge volume, the overall amount of synthetic musk discharge was still large. Therefore, stringent control on the wastewater discharge is advisable, as the cumulative effect of synthetic musks on the agriculture, fish farming and human health may still exist in the further [32].

#### 4. Conclusion

In this study, eight synthetic musks were successfully detected by using the method of ASE. An optimum experimental condition, solvent: n-hexane/acetone (1:1, V:V),

extraction temperature:  $100^{\circ}$ C, 5 min extraction time and 2 extraction cycles was obtained. The purification process was operated by in cell clean-up with Florisil. With this method, the LODs ranged from 0.03 to  $0.05 \text{ ng g}^{-1}$ , and the recovery rates ranged from 86.0% to 104%, the RSD was satisfactory for all sample analytes. When compared with SE and USE, ASE has the best extraction efficiency. Considering other factors such as solvent consumption, extraction time and operability, ASE is also preferable to SE and USE.

In 15 samples from Taihu lake, DPMI, ADBI, AHMI, ATII were below detection limit, and the HHCB, AHTN were detected in all of the samples, the concentration of the HHCB, AHTN, MX, MK were  $0.336-3.10 \text{ ng g}^{-1}$ ,  $0.184-1.21 \text{ ng g}^{-1}$ , BDL- $0.349 \text{ ng g}^{-1}$ , BDL- $0.0786 \text{ ng g}^{-1}$  dw, respectively. The concentrations of synthetic musks in sediment samples from Taihu Lake were lower than that of other areas of China, such as Zhujiang River, Suzhou Creek, but they have similar levels when compared with that of Lake Erie, Lake Ontario (Canada) and San Francisco Bay in USA. Considering the huge volume of Taihu Lake, it is still obvious that a large amount of synthetic musks were discharged into the lake. The potential hazards of synthetic musks may affect the agriculture, fish farming and even human health. More stringent measures should be taken to reduce the overall wastewater discharge into the lake.

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