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Monitoring, sampling, and automated analysis

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• Abstract

A review of the literature published in 2018 on topics related to monitoring, sampling, and automated analysis is presented. The review includes current developments in monitoring, sampling, and analysis of water, wastewater, and groundwater. This review includes the following sections: brief introduction; sample preparation and extraction techniques; real-time, high-frequency, and/or in situ monitoring (microbiological, inorganic, organic, metals, and others); passive monitoring; and the biosensors. In the end, the authors have discussed future of the topic. © 2019 Water Environment Federation

• Practitioner points

- Advances in monitoring, sampling and automated analysis of water and wastewater are summarized.
- Real-time, high-frequency, and in-situ monitoring and analysis of pollutants are summarized.
- Topics include sample preparation and extraction and passive monitoring, and biosensors for pollutants.

• Key words

biosensor; in situ monitoring; novel method; passive monitoring; real-time monitoring; sensor; water quality

Introduction

With advancement in analytical capability, sensing technology, computational power, and data storage space, monitoring, sampling, and automated analysis has seen huge developments recently. These advancements have led to a lower detection level, high throughput, high sensitivity, better speciation, and short analysis times. However, there still are limitations such as contamination, changes in composition during sampling and analysis, poor recovery, poor separation efficiency, interferences, low resolution, insufficient selectivity, and false positives and false negatives during automated analysis (Schmidt, 2018).

Sample preparation and extraction

A review article highlighted the properties and applications of graphene-based sorbent materials for online and offline sample preparation techniques, their synthesis, and modes of extraction (de Toffoli, Maciel, Fumes, & Lanças, 2018). Some of the techniques covered were solid-phase extraction (SPE), magnetic SPE, dispersive SPE, solid-phase microextraction (SPME), microextraction by packed sorbent, stir-bar sorbed extraction, online SPE, in-tube and online SPME, and online microdialysis.

Lorenzo-Parodi, Kaziur, Stojanović, Jochmann, and Schmidt (2018) published a review article on benefits, drawbacks, and applicability of several microextraction techniques, including SPME, stir-bar sorptive extraction (SBSE), solid-phase dynamic extraction (SPDE), and in-tube extraction-dynamic headspace (ITEX-DHS). The article focused on selection of suitable extraction methods for polyaromatic hydrocarbons, polychlorinated biphenyls, pesticides, short-chained chlorinated paraffins, and volatile organic compounds.

Raza, Hashemi, Kim, Lee, and Deep (2018) published a review article focusing on sampling and preconcentration methods for aromatic hydrocarbons in environmental matrices such as air, water, and soil.

In order to prevent sample evaporation, contamination, and gas exchange after sampling a water sample by an automatic sampler, a mobile autosampler that injects water samples directly into air-tight vials was developed (Hartmann et al., 2018). Compact dimension, low weight, and flexibility in sampling interval were identified as strengths of the sampler by the authors. The paper focused on long-term stability, mechanical functioning of the sensor, and potential applications.

Fabric fiber sorbent extraction platform was developed using polyster fabric fibers coated with sol-gel polydimethylsiloxane and used in an automatic SPE microcolumn preconcentration system to extract ultra-trace elements lead and cadmium and analyze in an atomic absorption spectrometry (Kazantzi, Kabir, Furton, & Anthemidis, 2018). Authors reported that the method was reproducible with high extraction efficiency and satisfactory sensitivity.

A sorbent material consisting of UVM mesoporous silica doped with titanium and iron was synthesized, characterized, evaluated, and validated for SPE of organophosphorus pesticides in water samples (Pellicer-Castell et al., 2018). The recovery, relative standard deviation, and limit of detection obtained were 81%–105%, 7.8%–12%, and 0.2–1.4 ppb, respectively.

A small-volume multiplexed pumping system (MPS) was developed to sample at a high frequency under volume-limited conditions (Maxwell, Birgand, Smith, & Aveni-Deforge, 2018). The sampling system coupled with enough premeasurement rinses reduced the volume of flow-through measurement cell from 40 to 1.1 ml. The application of the small-volume MPS was demonstrated by studying bioreactor and in-stream mesocosms.

Real-time, high-frequency, and in situ monitoring and analysis

Microbiological

Mycrobiota platform, based on culture-free 16S rRNA gene next-generation sequencing (NGS), was used to monitor microbial dynamics of a full-scale, nonchlorinated drinking water distribution system (Boers et al., 2018). Spatial and temporal (seasonal) changes in bacteria genus were successfully detected and identified in contrast to conventional methods.

Real-time bacterial monitoring was carried out in a reverse osmosis treatment plant using a real-time continuous bacterial counter (Fujioka et al., 2018). Bacterial counter was reported to be a viable method for tracing bacterial count in feed and the permeate during the reverse osmosis process by comparing with results from epi-fluorescence microscopy for total bacterial counts ($R^2 = 0.83$).

A smartphone-based in-gel loop-mediated isothermal amplification system was developed to quantify coliphage MS2 in environmental water samples within 30 min and tested in artificially spiked and naturally contaminated water samples (Huang et al., 2018). The method was based on immobilization of viral particle by reagents, amplification, and determination of fluorescent amplicon dots using a smartphone imaging. The method had similar sensitivity to quantitative reverse transcription polymerase chain reaction and was tolerant to inhibitors naturally present in water.

A method was developed to speed up the detection of somatic coliphages to 3.5 hr (Muniesa et al., 2018). The method consisted of incorporating a tailored *Escherichia coli* strain to produce blue color after infection of host strain cells.

Tryptosan-like fluorescence (TLF), a component of UV-fluorescent dissolved organic matter at excitation-emission wavelengths of 280 and 360 nm, was significantly correlated with thermotolerant coliform concentration in surface and groundwater (Sorensen, Baker, et al., 2018). Using a TLF threshold of 1.3 ppb, a 15% false-negative error and 18% false positive were obtained. The method was not suitable for <10 thermotolerant coliforms cfu per 100 ml. The authors claimed that the technology is inexpensive and viable for monitoring fecal contamination globally as commercial fluorimeters are easily available; can be used in remote environments; does not require consumables; and provides immediate reading regardless of common interferents.

Sorensen, Vivianco, et al. (2018) used fluorimeters to monitor microbiological quality of raw water in real time at the water intake in UK public water supplies and compared the results to discrete sample analysis of *E. coli*, total bacterial cell count by flow cytometery, and turbidity-based online measurements (Sorensen, Vivianco, et al., 2018). Fluorescence spectroscopy was based on dissolved organic matter target peak at excitation/emission wavelengths of 280/365 nm for TLF and 280/450 nm for humic-like fluorescence (HLF). Authors concluded that the correlationship of *E. coli* and total bacteria with TLF and HLF was strong (0.71–0.76), greater than that with turbidity (0.40–0.48).

According to Buysschaert, Vermijs, Naka, Boon, and De Gusseme (2018), flow cytometry can be used to monitor changes in freshwater communities automatically online. Bacteria were observed after every treatment step including reverse osmosis, and the bacterial concentrations were related to process operations in a full-scale water treatment plant. Results indicated that flow cytometry technique could be used to monitor membrane integrity and microbial quality of water. In another study, a custom-built continuous staining device, real-time flow cytometry data acquisition, and flow cytometry fingerprinting were used to quantify microbial communities in aquatic ecosystems (Props et al., 2018). At least 1,000 cells' measurement was required to accurately measure the concentrations.

Chan et al. (2018) investigated using flow cytometry to monitor the performance of biofilm function in a full-scale slow sand filters (SSF). Flow cytometry profiles were studied for three SSFs differing in microbial community maturation and sand composition before and after scraping events. Simultaneously, water quality including microbial and chemical parameters of influent and effluent water was studied and correlated with flow cytometry profiles. Authors reported that flow cytometry method was useful in rapid detection of dynamic microbial changes in the SSF biofilm and effluent water.

A study combined Raman microspectroscopy and metaproteomics to label active bacteria in groundwater by D_2O and monitor their metabolic activity and functional characterization in complex ecosystems (Taubert et al., 2018). Addition of non-labelled model compounds, e.g. veratric acid or methylamine, to the groundwater microcosms was found to be suitable for evaluating changes in metabolism of microbial population due to stimuli.

Inorganic

A sensor for real-time nitrate monitoring in water was developed based on electrochemical impedance spectroscopy (EIS) with ion-imprinted polymer (IIP) as a coating material (Alahi, Mukhopadhyay, & Burkitt, 2018). The sensor results were compared with commercial sensors and validated with UC spectrometric method. The results confirmed detection limit of 1–10 mg/l, error of <5%, and reusability up to five times before cleaning.

A colorimetric method based on the Griess reaction to detect nitrite in water using mobile phone was developed (Puangpila, Jakmunee, Pencharee, & Pensrisirikul, 2018). In Griess reaction, nitrite ion reacts with sulfanilic acid under acidic conditions to produce a diazonium salt that reacts with *N*-(1-nathtyl)-ethylenediamine dihydrochlorie to produce a red-violet solution. The developed method used LED flash lights to capture an image of red-violet solution using built-in camera and analysis of image by a program to correlate red color intensity with concentrations. The authors reported that they obtained 4% accuracy, <1% precision, and 0.03 mg/l detection limit.

In another study, infrared spectroscopy method for nitrite detection in water was modified by adding ion-exchange resin for enrichment and an FTIR spectrometer for measurement (Du, Ye, & Dong, 2018). The limit of detection achieved was 1.7 mg/l when 0.1 g resin and 60 min of adsorption time was used. The method was flexible due to possibility of changing mass of resin and adsorption time.

Two stochastic microsensors based on protoporphyrin IX and graphene were used to simultaneously analyze nitrate and nitrite in water samples (Stefan-van Staden, Mincu, van Staden, & Gugoasa, 2018). The method was reported to have recoveries >99%, RSD lower than 1%, and good correlation with ISO standard methods.

An in situ and real-time sensing platform for phosphate in natural waters was developed using microfluidics, colorimetric reagent chemistries, and LED-based optical detection (Donohoe, Lacour, McCluskey, Diamond, & McCaul, 2018). The platform was used in laboratory over 55 days for measuring concentrations of standard solutions and in the field over 5 days. The authors reported that the method was reasonably reliable (mean relative errors and standard deviation of 1%–2%, the limit of detection of 8.55 μg/l [0.09 μM]) compared to UV-Vis spectrophotometer reference method.

In another study, analysis of phosphate in water and wastewaters was performed by molybdenum blue method in a novel automatic flow system that integrated direct injection photometric detection with multipumping flow system (Koronkiewicz, Trifescu, Smoczynski, Ratnaweera, & Kalinowski, 2018). The analysis range and repeatability were 0.1–12 ppm and 2.2, respectively. Low consumption of reagents, low generation of waste, and high accuracy were reported.

A simple automated analyzer called integrated syringe-pump-based environmental water analyzer was designed to measure the ammonium in fresh water (Ma et al., 2018). The system consisted of a mini-syringe pump with a selection valve controlled by LABVIEW program to pump sample and reagents, and a spectrophotometer for detection. Authors reported that the results were robust and reliable with a detection limit of 0.12 μM, relative standard deviation of 0.2%–3.4%, a linear calibration curve with R^2 of 99.98% and validated by comparing to reference methods.

An ion-exchange-based inline pretreatment system was combined with high-performance liquid chromatography (HPLC) and a photochemical reaction and chemiluminescence detector (HPLC-PR-CL) to analyze *N*-nitrosamine (Kodamatani et al., 2018). Authors reported that the method did not require a pretreatment, is not interfered by other anions including nitrate, and is sensitive (LOD of 0.42–1.4 ppb). The results were validated against GCMS.

Organic

A microbial fuel cell sensor based on a gas diffusion biocathode sensing element made up of cultivated bioanodes and breathable membrane was fabricated and used for formaldehyde detection from 0.0005% to 0.005% in aerobic and anaerobic water environment (Jiang, Liang, Huang, & Ren, 2018). The developed sensor was used to monitor formaldehyde-contaminated air and water in aerobic environment for air and both aerobic and anaerobic environments for water. Authors reported that formaldehyde detection limit was 20 ppm and the sensor could be used for air and water pollution monitoring.

Scala-Benuzzi, Raba, Soler-Illia, Schneider, and Messina (2018) used electrochemical paper-based immunocapture assay (EPIA) to preconcentrate and detect emerging pollutant ethinyl estradiol in water. The assay is based on paper microzones modified with silica nanoparticles and anti-ethinyl estradiol-specific antibodies for preconcentration. The ethinyl estradiol was desorbed from preconcentrated paper microzones electrochemically in dilute solution of sulfuric acid with screen-printed carbon electrode modified with reduced graphene as electrode. The recovered ethinyl estradiol was then electrochemically detected by Osteryoung square wave voltammetry. The limit of detection was 0.1 ng/l, and linearity and recovery were acceptable.

A rhodamine-based probe-containing α-bromoamide was used to detect glutathione, an essential thiol in the cell for physiological functions, in both a colorimetric and on/off fluorescence probe (Chen, Park, Wu, Kim, & Yoon, 2018). Authors applied the sensors to sense endogenous and exogenous glutathione in HeLa cells and glutathione in zebrafish for a high olfactory pit tissue.

Jonsson, Paulsson, and Kreuger (2018) developed time-integrating, microflow, in-line extraction passive sampler to extract pesticides and other organic pesticides by passing water first through a flow restrictor using a rubber band-driven pump and SPE cartridges. The SPE cartridge was then extracted and analyzed online using LC-MS/MS. The results were compared with grab samples. Authors reported that total of 72 pesticides at environmentally relevant concentrations with a limit of detection of 0.1–1 ng/l were analyzed.

A method to monitor naphthalene, a representative polyaromatic hydrocarbon, in water in the presence of suspended organic matter was developed by modifying fluorescence spectroscopy (Mbaye et al., 2018). The method consisted of scattering correction based on excitation beam scattering due to suspended organic matter and fluorescence correction based on modeling of remaining signal to estimated napthalene content.

A fully automated analysis method for 37 legal and illicit psychoactive substances including cocaine-related compounds, hallucinogens, opiods, cannabinoids, stimulants, benzodiazepines, antidepressants, sedatives, antipshychotics, and hypnotics in wastewater was developed based on online SPE and liquid chromatography tandem mass spectrometry (López-García, Mastroianni, Postigo, Barceló, & López de Alda, 2018). The method was validated based on linearity, recovery, repeatability, and sensitivity. The limit of detection was 7–23 ng/l for most compounds. A week of real wastewater sample analysis detected 25 of 37 compounds in the wastewater in Barcelona, Spain, with an average concentration of 7–54 ng/l.

Analytical method for diclofenac in water samples was developed using vortex-assisted liquid–liquid microextraction and subsequent GCMS analysis (Aydin, Aydin, Beduk, Tekinay, & Kilic, 2018). Use of 200 μl chloroform, 25 μl *N*-methyl-*N*-trimethylsilyl-trifluoroacetamide, vortex extraction, and derivatization at 3,000 rpm for 5 min was considered optimal conditions. Lower organic solvent consumption and better recovery than SPE were reported as positives of the method.

A method to detect 12 perfluoroalkyl acids in water samples using online SPE and HPLC-MS/MS was developed (Barreca et al., 2018). The method detection limit was reported as 0.2 ng/l.

Metals

A review paper on development of new nanomaterials and current use of nanomaterials for speciation analysis of toxic elements, including mercury, arsenic, chromium, and selenium, was published (Xu, Hu, Zhang, Hou, & Jiang, 2018).

A Fe^{+3} sensor based on 1,4-diketo-3,6-diphenylpyrrolo[3,4-c]pyrrole and colorimetric and fluorescent turn-on was developed and tested in aqueous dimethyl sulfoxide media and environmental water samples (Zhang, Sun, et al., 2018). Color change from purple to red allowed naked-eye detection. The lower detection limit, recovery, and relative error were 14.3 nM, 93%–104%, and 2.8–7.4, respectively. Authors reported that the method showed high anti-interference performance, good versatility, wide pH response range, and instantaneous response time.

A study to investigate the use of complex permittivity to monitor lead in the soil found that the complex permittivity method has potential to be used as nondestructive method to monitor lead in the contaminated soil (Martini & Shang, 2018).

Others

Isotopic variation in irrigation water and hydrological processes in rice fields was investigated in situ using automatic water sampling system and isotopic analysis of water on a subhourly basis (Mahindawansha, Breuer, Chamorro, & Kraft, 2018). The automatic analytical system was based on water-level gages, low-cost car wiper pumps, and a control system with python controller to sample every 30 min, and the isotopic analysis was carried out with spectroscopy. Subdaily variation in isotopic signature in ground and surface water was successfully studied.

In a study, a treatment system for rainwater or groundwater with feedback control was designed to ascertain the quality of the treated water (Guzman, Josue, Raynes, & Purio, 2018). The treatment system consisted of the storage tank, pumps, treatment unit, sensors, microcontroller, and treated water storage tank. The authors used feedback mechanism to re-route the treated water to the influent tank until the parameters (pH, conductivity, redox potential, turbidity, and temperature) measured by the sensor were within the specified range.

Zhang, Liu, et al. (2018) proposed conductivity monitoring-based estimation to quantify rainfall-derived inflow and infiltration (RDII) in a sanitary sewer when RDII and overflow occur at the same time to improve flow-based estimation. The method consisted of using Fourier transformation to analyze variation in flow and conductivity during dry weather and nonlinear curve fitting. The resulting method was used to estimate RDII during three rainfall events of different intensities.

Sulfide species in water react with *N*, *N*-dimethyl-phenylenediamene sulfate in the presence of Fe^{+3} to form fluorescent methylene blue species. This fluorescence signal was used to measure sulfide concentration from 0 to 10 mg/l with a detection limit of 0.5 mg/l (Petralia et al., 2018). A linear fit with $R^2 = 0.981$ was obtained during correlation with UV-Vis detection method.

Passive monitoring

In situ passive monitoring using the diffusive gradients in thin films (DGT) technique was developed for six frequently studied organophosphorus flame retardants (Zou et al., 2018). The DGT devices contained hydrophilic–liphophilic balanced (HLB) resin in agarose gel as binding agent. Comparable concentrations to grab sampling were obtained using the developed DGT technique, and the capacity of the binding gel was >115 μg per disc and did not deteriorate over 131 days. The developed method was able to measure up to ng/l levels under diverse environmental conditions including pH, ionic strength, dissolved organic matter, deployment time, and diffusive layer thickness.

Castle et al. (2018) analyzed metaldehyde in surface water using a "Chemcatcher" passive sampler that was made up of a hydrophilic–lipophilic balanced (HLB-L) disk as the receiving phase underneath a polyethersulphone membrane. Sample uptake rate was evaluated at the laboratory and found to be within acceptable range. Authors calibrated and evaluated at the field for real water samples and found the results to be complementary to grab samples.

Tylosin, sulfamethazine, and atrazine were monitored in tile-drain effluent and surface water using polar organic integrative sampler (POCIS), a passive sampling technology (Washington, Moorman, Soupir, Shelley, & Morrow, 2018). The results from POCIS were used to evaluate occurrence and temporal variation of the selected antibiotics.

Biosensor

A review article on electrochemical biosensor with a focus on its use to detect various types of contaminants including heavy metals was published (Hernandez-Vargas et al., 2018).

An optical biosensor based on the symbiosis between the alga *Chlorella vulgaris* and the ciliate *Tetrahymena pyriformis* and measurement by fluorimeter was developed to monitor marine pollutants online (Turemis et al., 2018). The biosensor was used to examine one or more pesticides: simazine, atrazine, and diuron, and the results were compared with the analysis by liquid chromatography mass spectrometry (LCMS). The limits of detection were observed in the range for 0.13–1.35 ppb for each pesticide. The authors claimed that the biosensor was simple, cheap, and rapid and required less maintenance.

A yeast-based biosensor was designed to screen simozine herbicide in spiked and real drinking water and cow milk samples (Grasso, Caracciolo, Cocco, Frazzoli, & Dragone, 2018). The probe was based on yeast oxygen consumption change due to toxicity of the simozine. Authors reported that concentrations up to 0.02 ppb in drinking water and 2 ppb in cow milk were measured.

Future of the topic

While this paper covered the recent development in diverse areas such as culture-independent rapid and reliable microbial method, compound-specific stable isotope analysis, automation of analytical methods and sample preparation, reduction in cost of analysis, in situ methods, and smartphone-based methods, these areas still need further development. These recent developments have to move toward the real-time and high-frequency monitoring application by agencies than demonstration at the research stage. Other research needs include lowering detection level; increasing the accuracy, recovery, and speciation; decreasing analysis time; decreasing contamination and changes in composition during sampling and analysis; and monitoring a target compound in the complex mixture of many compounds.

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