

Real-Time Water Quality Monitoring with Chemical Sensors

Subjects: Water Resources

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Water quality is one of the most critical indicators of environmental pollution and it affects all of us. Water contamination can be accidental or intentional and the consequences are drastic unless the appropriate measures are adopted on the spot.

Keywords: water quality ; real-time monitoring ; multisensor system

1. Water Quality Monitoring Systems

The first sensor really suitable for water monitoring was the glass pH electrode, which appeared, in the present shape, along with a pH meter, around 1930. Since then, pH is a primary parameter of most water monitoring devices.

It is obvious, however, that multiple water parameters must be evaluated to responsibly judge its quality and multisensor systems should be applied for such purposes.

There have been multiple attempts to develop multisensor systems that could be applied for water quality control, e.g., ^[1] ^[2]. However, the first efforts mostly dealt with laboratory water analysis rather than being applied in real-time, online mode.

For instance, a voltammetric sensor array with four electrodes (Au, Pt, Ir and Rh) served for multisite water quality monitoring at a water treatment plant ^[3]. The aqueous samples were taken at nine filtration steps, as well as before and after the complete procedure of water purification. The voltammetric data were processed by principal component analysis (PCA), revealing pronounced difference between raw, rapidly filtered and clean water. However, it was observed that the samples collected after treatment by several slow filters were close to the rapidly filtered water samples on the PCA scores graph. This can potentially be explained by the low efficiency of these filters. Therefore, it was concluded that a multisensor system approach is suitable for continuous control of water quality at treatment facilities, indication of the possible malfunctioning units and for checking the water status after maintenance. Significant influence of sensor drift and the necessity to compensate this drift was pointed out.

The system developed in ^[4] was designed to measure pH, temperature, dissolved oxygen, conductivity, redox potential and turbidity. This set of parameters is the most common one in water quality assessment since the sensors for these parameters can run in continuous mode. The whole set of sensors was mounted on aluminum oxide. All sensors were united into a single PVC body and their outputs were collected by the data acquisition system, which could also perform remote data transmission. The work suggests that the body might be dipped into water or even built into water flow. The device also included a set of electric valves and pumps for sampling, cleaning and calibration. The authors proposed that such a portable system can be suitable for water quality monitoring from different sources.

Chinese authors published a paper where a multisensor system was applied for the determination of several elements such as iron, chromium, manganese, arsenic, zinc, cadmium, lead and copper ^[5]. The device comprised three analytical detection systems: a multiple light-addressable potentiometric sensor (MLAPS) based on a thin chalcogenide film for simultaneous detection of Fe(III) and Cr(VI) and two groups of electrodes for detection of other elements using anodic and cathodic stripping voltammetry. The following detection limits were obtained: Zn—60 µg/L, Cd—1 µg/L, Pb—2 µg/L, Cu—8 µg/L, Mn—60 µg/L, As—30 µg/L, Fe—280 µg/L and Cr—26 µg/L. The authors recommended their method to determine metals simultaneously in seawater and wastewater; however, the possibility of application of this device for online analysis was unclear.

Potable water quality is of primary interest to people. Such type of water was studied in [6], using two sensor stations. The first one was used for detecting free chlorine with a precision of 0.5% and limit of detection (LoD) of 0.02 mg/L, as well as total chloride by colorimetric method with precision of 5% and LoD 0.035 mg/L. The second station used was a multisensor for detection of pH, redox potential, dissolved oxygen, turbidity and conductivity. Eleven different contaminants were injected into the flow of the studied liquid, namely pesticides, herbicides, alkaloids, E. coli, mercury chloride and potassium ferricyanide. It was demonstrated that the set of sensors produces a response for each type of contaminants. Unfortunately, the work does not report any data about the precision of such systems during long-term application.

Two multisensor systems were suggested in [7] for environmental monitoring of various contaminants. One was dealing with contents of ammonium, potassium, and sodium in a river with low anthropogenic load. The second system was installed in river water in a populated region and was designed for detection of heavy metals such as copper, lead, zinc and cadmium. The systems were also equipped with radio transmission devices. The system was tested just for 8 h, which is obviously too short a period for any serious conclusions about such a technology.

Wider research was performed in [8], which was conducted over a period of 12 months. A sensor array comprising eight conducting polymer sensors for gas phase analysis was used to detect abrupt changes in the wastewater quality. Free gas emanating from bubbled liquid in the flow cell with constant temperature was delivered to the sensor chamber for analysis. The results of field tests at the water treatment plants using automatic systems produced water quality profiles and displayed the possibility of determining both random and model contaminants. This approach showed high sensitivity and flexibility and low dependence on long-term drift, daily oscillations, temperature and humidity. It must be noted, however, that the described experiments were carried out not at a real water treatment plant, but in a pilot system. Thus, the diversity of its performance may not be representative for real-world conditions. Besides, the idea to follow water quality via headspace analysis is obviously limited: it is impossible to follow contaminants which are not volatile enough.

2. Application of Biosensors and Optical Sensors for Water Quality Assessment

Biosensors were also used for water quality control, though quite a few of them were applied for online flow analysis. Pesticides were the main target of biosensors.

A system of biosensors capable of determining dichlorvos and methylparaoxon in the water was suggested in [9]. The systems consisted of three amperometric biosensors based on various AChE (acetylcholinesterase) enzymes. These enzymes were immobilised in a polymeric matrix onto the surface of screen-printing electrodes. The enzymes solutions were deposited over the electrode surface and irradiated by light, inducing photo polymerisation of the azide groups in the molecules. Such a sensor array was built into a flow system permitting automatic analysis. Bottled and river water was studied. The concentration of pesticides was detected in the ranges 10^{-4} – $0.1 \mu\text{M}$ for dichlorvos and 0.001 – $2.5 \mu\text{M}$ for methylparaoxon. Solely spiked samples were considered; therefore it is necessary to further verify performance of such a system in online mode.

Another work [10], reports on using Pt electrodes instead of screen-printed ones and self-made carbon paste was applied as a sensing layer. The paper implies that such a procedure may improve sensitivity of the substrate for some of the immobilized enzymes. The total number of biosensors in the array was eight. The ultimate aim of this research was not a quantification of pollutants but a global evaluation of water quality. It is doubtful though, if such a quality can be precisely determined by biosensors, which are highly selective to the main substance and would exhibit low cross-sensitivity to many other analytes present in the natural water.

One more attempt to evaluate global water toxicity by biosensors is described in [11]. The online toxicity monitoring system employed sulfur oxidizing bacteria (SOB) and consisted of three reactors. No toxicity changes in the natural flow water were observed over a period of six months. When the flow was spiked with diluted pig farm waste, the activity of sulfur oxidizing bacteria decreased by 90% in 1 h. The addition of $30 \mu\text{g/L}$ of nitrite ions or $2 \mu\text{g/L}$ of dichromate ions resulted in full degradation of sulfur oxidizing bacteria activity in 2 h. Thus, the sensitivity of the system to both inorganic and organic pollutants was demonstrated. It must be noted that one or two hours is a rather long period of time for detecting acute contaminations; functionality of the system could be regained only by introducing a new portion of bacteria, which significantly impairs real-time, online application of such device.

Optical sensors were also recently applied for water quality analysis, however, these are mostly discrete sensors, though tuned sometimes for integral parameters such as water color, turbidity or even COD and BOD. Discrete sensors were

used to determine chlorophyll in the seawater on the basis of its fluorescence [12], for evaluation of water opacity and color evolution by LED [13], for analysis of water turbidity and color in online mode [14] as well as for determination of heavy metal ions [15].

3. Biomimetic Approaches for Sensing Water Quality

3.1. Chemical Sensors for Sensing in “Real-Life Environments”

Biosensors reveal exceptional selectivity and often sensitivity, but usually are limited in terms of ruggedness and technical applicability in non-physiological conditions. One way to overcome this is to implement bioanalogous selectivity into systems that are able to withstand harsh and non-physiological conditions, so-called biomimetic systems [16]. Molecularly imprinted polymers (MIPs) are a promising example of such synthetic materials [17], since they are robust due to their highly cross-linked nature. Furthermore, they come at much lower costs than natural materials and provide longer storage and use periods. MIPs can also be produced for molecules that cannot be detected by natural receptors [18].

MIPs are generally synthesized by co-polymerization of functional and cross-linking monomers in the presence of a template (see **Figure 1**). Initially, a complex forms between functional monomers and the template through weak, noncovalent interactions (mainly hydrogen bonds, Van-der-Waals or π - π interactions), followed by polymerisation with cross-linking monomers to form a rigid, three-dimensional polymeric network. Removal of the template leads to recognition sites (cavities) within the polymer that are complementary to the target molecule in size, shape, and chemical functionality and are suitable to selectively rebind the analyte [19].

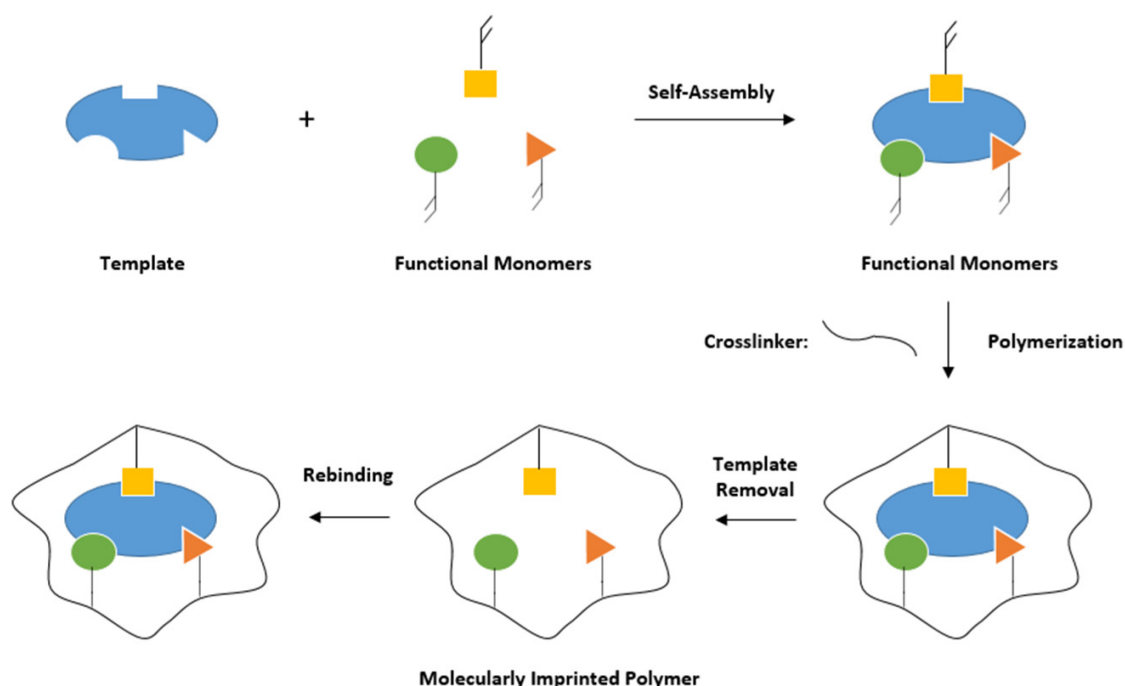


Figure 1. Schematic overview of molecular imprinting.

Except for MIPs, target recognition can also be obtained using other strategies. Aptamers, for example, are single-stranded RNA or DNA oligonucleotides, whose tertiary structure selectively binds their target molecules [20]. Another option is whole-cell-based sensors, which were also already applied to real wastewater samples [21][22]. In this case, mammalian cells were used for detecting harmful and toxic compounds, because their closeness physiology is close to that of humans. Although this strategy may not be regarded biomimetic in the strict sense of the word, it provides direct information about the overall toxicity of samples rather than detecting or quantifying one specific substance. When applying this method, unknown or new chemicals and pollutants may be detected. Kubisch et al. used rat myoblast cells in combination with a commercially available multiparametric readout system to measure impedance (morphological integrity) and two metabolic parameters—acidification and respiration—to investigate the overall toxicity and bioavailability of substances in water samples [21]. This was achieved by using three different types of electrodes on a single chip surface: impedance, pH and oxygen (CLARK) electrodes. After testing different test compounds, including metal ions and neurotoxins, the system was exposed to real wastewater samples. It responded to different contaminants and was indeed suitable for monitoring unknown, harmful compounds in water. Similarly, the group of C. Guíjarro applied rat liver cells in a whole-cell-based sensor system to monitor environmental contaminants, including an insecticide and a flame retardant, in water samples using the same analyzing system [22].

The aforementioned advantages of MIPs make them an attractive tool for different applications, such as solid phase extraction, drug targeting, development of sensors for various types of analytes, and environmental monitoring. Although the number of publications concerning MIP-based sensors is rising, only a small amount is actually applied to real-life environments or complex matrices. This part of the entry will provide an overview of chemical sensors that were tested in (real-life) water samples with a special focus on receptor layers based on MIPs.

3.2. MIP-Based Sensors for Water Analyses

In 2018, Ayankojo et al. introduced a sensor system capable of detecting pharmaceutical pollution in aqueous solutions [23]. They chose amoxicillin as the model analyte and implemented a hybrid MIP, consisting of organic and inorganic components, on the gold surface of a surface plasmon resonance (SPR) transducer. The hybrid MIP film was synthesized by applying the sol-gel technique and using methacrylamide as organic monomer and vinyltrimethoxysilane as inorganic coupling agent to form a stable and rigid polymeric network. Sol-gels have a highly porous structure and recognition sites are usually formed in a more ordered way. This results in enhanced sensitivity and faster sensor response times. Rebinding experiments of the amoxicillin MIP in phosphate-buffered saline (PBS) and tap water revealed an imprinting factor of 16 compared to the nonimprinted polymer (NIP) and a limit of detection $LoD = 73 \text{ pM}$. Furthermore, the MIP responded almost exclusively to its target analyte thus exhibiting utmost specificity. In the same year, the group of Cardoso also developed a sensor for detecting chloramphenicol, an antibiotic used in fish farms [24]. The corresponding MIPs were electro-polymerized on screen-printed carbon electrodes.

Impedance and square wave voltammetry (SVW) in both electrolyte solution and water from a fish tank served to investigate the performance of the recognition element. In case of impedance measurements in electrolyte solution, sensor characteristics were linear in a concentration range from 1 nM to $100 \text{ }\mu\text{M}$, achieving an $LoD = 0.260 \text{ nM}$; SVW yielded similar characteristics and an $LoD = 0.653 \text{ nM}$. In real-life samples—water from a fish tank—sensors responded linearly down to 1 nM and achieved an LoD of 0.54 nM and 0.029 nM for impedance and SVW measurements, respectively. These results suggest that there is no significant impact on sensor behavior when switching from standard solutions to real water samples leading to reproducible and sensitive sensor characteristics over five orders of magnitude down to 1 nM .

The real-life feasibility of MIP-based sensor systems were also demonstrated in case of detection of faecal contamination of seawater samples [25]. MIP nanoparticles were fabricated for sensing *Enterococcus faecalis* (*E. faecalis*) serving as faecal indicator to assess the water quality. Such MIP nanoparticles have the advantage of a higher surface-to-volume ratio, which means that the resulting cavities or binding sites are easier to access by target analytes [26]. *E. faecalis*-imprinted nanoparticles demonstrated good SPR sensor performance in aqueous and real seawater samples:

Khadem et al. fabricated an electrochemical sensor for detecting diazinon, an insecticide, based on a modified carbon paste electrode combined with MIPs and multi-wall carbon nanotubes (MWCNTs) [27]. Using the latter modifier improves conductivity, whereas MIPs offer the necessary sensitivity towards the template molecule. After optimizing electrode composition, the method was first validated in aqueous standard solutions. SVW measurements revealed that the MIP showed much higher affinity to the analyte than the reference, the nonimprinted polymer; the system achieved linear performance in the concentration range from 5×10^{-10} to $1 \times 10^{-6} \text{ mol/L}$ with a calculated $LoD = 1.3 \times 10^{-10} \text{ mol/L}$. Furthermore, it was considerably more selective to the analyte than to other tested substances (ions and other pesticides). To investigate the applicability of the system to real biological and water samples, different amounts of diazinon were spiked to urine, tap and river water. In all these cases the sensors detected the target analyte with high recovery rates ($>92\%$). This work demonstrates the use of MIP-based sensors in real-life samples and environments without the need of special sample pretreatment or preconcentration steps.

Another example for pesticide detection is presented in the work of Sroysee et al. [28]. They developed an MIP-based quartz crystal microbalance (QCM) sensor for quantification of carbofuran (CBF) and profenofos (PFF). For that purpose, an in-house-developed dual-electrode system was used, where one electrode pair served as reference with the upper electrode being coated with the NIP. Doing so offers the advantage of measuring MIP and NIP simultaneously under the same conditions. Applying the bulk imprinting method, MIPs for PFF were based on polyurethanes whereas CBF MIPs were synthesized using acrylic monomers. Frequency measurements of MIP- and NIP-coated QCMs are shown in **Figure 2**.

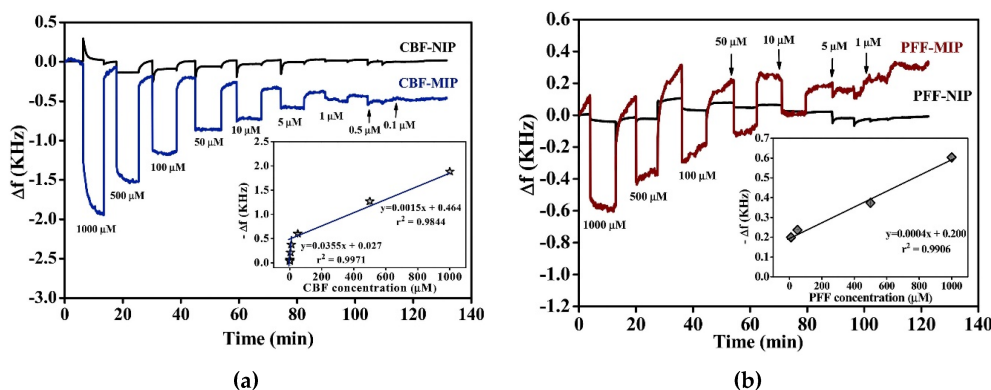


Figure 2. Frequency measurements of MIP- and NIP-coated QCMs for detection of (a) CBF and (b) PFF at different analyte concentrations. Reproduced from [28] Creative Commons License CC BY-NC-ND 4.0.

One can clearly see that both CBF and PFF MIPs led to linear sensor responses between 0.5–1000 μM and 5–1000 μM for CBF and PFF, respectively, whereas the frequency signal of the NIP stayed more or less constant.

Polycyclic aromatic hydrocarbons (PAH) are organic compounds which consist of at least two condensed aromatic rings. They are released into the environment through incomplete combustion of organic materials and considered to be mutagenic and carcinogenic. They usually occur in mixtures and their concentrations in air, water and sediments can be very low. Therefore, detection systems for PAH analysis need to be sensitive and selective. In particular, fluorescent sensors based on MIPs have gained in popularity due to their advantageous properties, such as high specificity, sensitivity and reversibility. Having a linear concentration dependency and low LoDs, those sensors seem to be quite promising for rapid detection of PAHs in aqueous solutions [29].

Sensors for the detection of nutrient components have been developed as well. For example, Warwick et al. reported a detection system based on MIPs combined with conductometric transducer for monitoring phosphates in environmental water samples [30]. Previous studies demonstrated that N-allylthiourea was the appropriate monomer for phosphate recognition [31]. The thiourea-based MIP was first optimized in terms of the optimal cross-linking monomer and ideal ratio of functional monomer to template (phenylphosphonic acid). Of all cross-linking monomers that were tested, ethylene glycol dimethacrylate (EGDMA) had the highest capacity of retaining phosphate as well as a monomer to template ratio of 2:1. After optimization, MIP membranes were integrated into the conductometric measuring cell. Selectivity tests in laboratory samples revealed no cross-talk to other ions, nitrate and sulfate. Both types of samples—standard and real-life ones—led to a linear increase in conductance with increased phosphate concentrations. In wastewater samples spiked with different amounts of potassium phosphate, the system allowed for LoD and LoQ values of 0.16 mg/L and 0.66 mg/L, respectively. The maximum acceptable amount of phosphate in wastewater is 1–2 mg/mL. The implemented sensor system with a linear range from 0.66 to 8 mg/mL therefore seems very promising for detecting small amounts of phosphate in environmental samples.

3.3. MIP-Based Sensors for On-Site Applications

For a pollutant sensor to be applicable on-site in real-life environments, it needs to be selective, reusable, robust and able to withstand harsh conditions. In 2015, Lenain et al. [32] reported a sensor that met all those criteria: it consists of spherical MIP beads deposited onto the electrodes of a capacitive transducer via electro-polymerization. Corresponding MIP beads for detecting metergoline—a model analyte for small molecules such as insecticides and pharmaceuticals—were synthesized through emulsion polymerization.

The sensor was also able to withstand harsh environments and achieved both a low LoD (1 μM) and low cross-selectivity. All these results suggest its suitability for monitoring pollutions originating from substances like pesticides or antibiotics in water samples (rivers, seawater) on-site.

Another example of a method suitable to the monitoring of contaminants in water *in situ* was introduced by Cennamo et al. [33]. It consists of an SPR sensor with an integrated plastic optical fiber (POF) combined with MIPs for detecting the model analyte perfluorobutanesulfonic acid (PFBS). With an LoD of 1 ppb, an interface software and the ability to connect to the internet directly, the SPR-POF-MIP technique is inherently suitable to detect small concentrations of different toxic or harmful compounds in real water samples *in situ*. Other advantageous features such as its reduced size, robustness and remote sensing abilities are further key factors for industrial applications of MIP-based sensor systems.

3.4. Mass Production of MIP-Based Sensor Systems

To prove that MIP-based sensors are inherently suitable for mass manufacturing, Aikio et al. developed a low-cost and robust optical sensor platform based on integrated Young interferometer sensor chips, where waveguides were fabricated on top of a carrier foil via roll-to-roll manufacturing techniques [34]. For chemical sensing of melamine, MIPs were used as recognition materials, whereas for biosensing of multiple biomolecules, sensor chips were functionalized with antibodies. In case of melamine sensing, the change in phase depended on the analyte concentration: Sensor responses increased with higher concentrations. Furthermore, the reference (NIP) led to much lower phase changes compared to sensor responses of the MIP. However, injection of higher concentrations (>0.5 g/L) led to saturation effects. For multianalyte biosensing, the sensor chip was functionalised with antibodies for C-reactive protein (CRP) and human chorionic gonadotropin (hCG) via inkjet printing. The results indicated that the Young interferometer bearing a specific antibody indeed selectively detected its corresponding protein. This work demonstrated the use of large-scale production techniques to develop a cost-efficient and rugged sensor system.

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