

## Sensor Methods for the Detection of Polycyclic Aromatic Hydrocarbons (PAHs) in Industrial Wastewater

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The adverse effects of industrial wastewater are well-known for deteriorating the quality of life through producing and releasing hazardous pollutants like polycyclic aromatic hydrocarbons (PAHs) into the water bodies. This review describes different sensing approaches for detecting and monitoring PAHs (such as phenanthrene, anthracene, fluoranthene, acenaphthene, chrysene, pyrene, and perylene) in industrial wastewater. Sensor methods are the most feasible and widely accepted approach for detecting and analysing PAHs in industrial wastewater. The sensor's performance is compared to alternative methods such as high-performance liquid chromatography (HPLC) and gas chromatography & mass spectrometry (GC-MS). The relative performance of different sensor-based detection methodologies is evaluated in essential quality assurance. Also, it highlights current limitations and future developments in the methodological approaches for their detection.

**Keywords:** PAHs, Industrial wastewater, Sensor method, Performance evaluation.

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### 1. INTRODUCTION

PAHs are mainly considered persistent, widespread, bio-accumulative, and toxic contaminants due to their anthropogenic, mutagenic, and carcinogenic nature and are found in industrial wastewater [1]. The increasing number of contaminants in industrial wastewater has raised a severe environmental concern [2]. PAHs (e.g., anthracene) are very harmful to human health and are responsible for various diseases such as lung, lymph, liver, and skin disorders [3].

In light of the harmful impacts of PAHs, there has been a great demand for environmental monitoring technologies for their accurate measurements. European Water Framework Directives such as Directives 2006/118/EC, 2000/60/EC, and 2006/11/EC have given prime importance to analytical methods which are very suitable for on-site detection and sensitivity toward wastewater pollutants [4]. The existing instrumental techniques (such as GC-MS, HPLC, etc.) [5] used for the identification of PAHs suffer from several demerits such as high cost with the demand for a higher amount of organic solvents, complex sample preparation, the complicated solvent extraction process, and time-consuming operation [6]. Regarding such limitations in conventional methods, the chemical sensor has proved to be a powerful analytical technique for evaluating particular analytes like PAHs [7].

A novel chemical sensor is one of the highly effective analytical techniques with many advantages like minimal waste production, online detection, and enabling on-site analysis. All of which should help contribute to green chemistry [8]. Based on the electrochemical properties of PAHs, a wide range of sensors has been developed, which are acceptable for detecting hydrocarbons

in air samples [9]. The optical sensor technique is based on various principles and their optical signals, such as absorbance, Raman dispersion or luminescence emission, and reflectance (fluorescence, chemiluminescence, and phosphorescence). In general, reflectance signals (fluorescence, phosphorescence, or chemiluminescence) are emitted through luminescence sensors. These signals are measured in convenient solid support after the immobilization of the analyte, providing a root for the expression of solid-phase luminescence (SPL). It also correspondent to solid-matrix luminescence (SML). The quantitative analysis of analytes is performed through analytical signals corresponding to the concentration of analyte in the sample. Warner's research group published an advancement in sensors and solid-surface luminescence methods [10]. Optical sensors coupled with flow injection analysis (FIA) for industrial samples, biomedical, and environmental were reviewed by Bosch and Sanchez [11]. In this review, they explained the importance of the optical method for chemical analysis and its applications in industrial process control, biomedical sensing, and environmental monitoring. Bosch and Sanchez also emphasized FIA [11], an automatic analytical method, for the quick analysis of the samples. FIA has gained much importance due to its low cost, simplicity, and ease of assembling analyzing set-up with a versatile analytical approach. In the present scenario, the flow-based luminescence sensors have attracted more for wastewater analysis, including desirable organic and inorganic contaminants [12].

A biosensor is based on luminescent bacteria and other essential monitoring apparatuses to detect organic pollutants, heavy metals, and other compounds in waters [13]. An analyte is restrained by adsorption, entrapment, or binding. Analyte retention happens

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through direct deposition on a flat surface or with the help of a syringe extraction procedure [14]. Microbeads immobilization technique is carried out to retain analytes [15]. Simple plane surface supports are used in the process, generally consisting of filters: papers and membranes. The membranes contain materials like polymeric membranes, nylon membranes, and C18-disks. In contrast, microbeads are comprised of silica gel (bonded-phase), ion-exchange resins, polymeric materials, etc. The disadvantages of working with microbead configurations are longer analysis time per sample and a required specific skilled operator.

Luminescence detection techniques such as phosphorescence, fluorescence, and chemiluminescence, as well as synchronous fluorometric detection methods, are used for water and environmental monitoring analysis problems [12]. Molecular imprinted polymers (MIPs) are a new recognition technique, since selective sorbents have attractive properties [16]. However, MIPs are only feasible for online monitoring systems consisting of small organic pollutants in waters [17]. Membrane-based conductometric sensors have been reported for several target compounds [18]. A conductometric sensor is also used for PAH detection considering MIP as the recognition layer [12]. The imprinting effect primarily relies on Vander Waals forces between self-organizing polymers and templates. The performance of small molecules having no functional group like PAHs is managed by non-covalent imprinting. A conductometric sensor has been developed based on MIPs, having 1.3 nmol/l anthracene detection limit which is allowable as it falls within PAHs permissible limit (3.93 nmol/l) as per WHO (World Health Organisation) guidance [19].

PAHs are broadly distributed in the environment, making it difficult to degrade naturally [20]. GC, GC-MS, and HPLC coupled with fluorescent spectrometry [21] are the most commonly used detection methods with accuracy. However, they require a long and heavy pre-concentration step for GC or LC analysis, which is not friendly to employ in field analysis. Also, they require expensive instruments, a long-time duration for signals acquisition and their analysis. For PAH detection, real-time analysis, field assay, high sensitivity, rapid data acquisition, and economical methods are required [22].

The electrochemical [23] and spectroscopic (for example, surface-enhanced Raman spectroscopy (SERS) [24] methods have been proposed for PAH identification in situ forms. Among the proposed methods, SERS is a promising method due to its high detection sensitivity as well as ample molecular fingerprint information [25]. Also, SERS has an extremely high level-up potential which converts structural analytical tools into a sensitive single structural molecule probe through Raman spectroscopy [26]. The optimum spatial zone is in the range of 0-4 nm where strong electromagnetic fields are localized for targeting strong Raman scattering [27].

In SERS detection, analytes should have a strong affinity for the substrates (primarily noble metals like Au and Ag) [28]. But PAHs have a poor affinity for metal surfaces, soluble in organic solvents, and are

unable to dissolve in water, which makes it disadvantageous to use the SERS detection method. Therefore, PAHs should be separated and enriched for the proper implication of the SERS detection technique. As a result, several modified substrates have been enhanced for the adsorption of PAH molecules onto the metal surface for label-free SERS detection. Such as, by applying a magnetic field, Ag and Au were immobilized and functionalized on the surface of magnetic micro- or nanoparticles which were later used for the separation of PAHs from the solution [29]. Other substrates of various functional entities were also proposed that includes thiol groups [30], Zhao group [31, 32], calixarenes [33], di-carbamates [34], viologen [35], cyclodextrin derivatives [32], and humic acids [36]. Some other proposed substrates are Calix [37], arene-Ag NPs functionalized with arene [38], dithiocarbonate functionalized Ag NPs, and 1-decanethiol monolayer assembled silver film which is transferred for PAH analysis via SERS detection [34]. These methods are highly sensitive to PAH detection through SERS. If more excessive modified reagents are used with nanoparticles functionalized group, then they can adsorb molecules of PAH more effectively. But at the same time, it may result in lowering the sensitivity of SERS detection. Also, it is very hard to separate these agents using a centrifuge process without affecting nanoparticle aggregation [22].

In the current years, researchers have focussed on substrates with particular geometries such as Novel SERS substrates with Au coffee ring [39], Au on TiO<sub>2</sub> nanotube arrays [41], Au on nickel 3D foam [40], Ag NPs deposited on SiO<sub>2</sub> [42], Au NPs on porous polymer [22], and alginate gel supported Au NPs [43], etc. for the detection of PAHs. Authors have also proposed a SERS platform for the screening of 16 PAHs (EPA priority based) composed via Fe<sub>3</sub>O<sub>4</sub> magnetic microspheres and Au NPs [44]. Rapid detection and a highly sensitive technique were proposed for SERS detection of anthracene, pyrene, phenanthrene, and their mixtures using optimization of Au NPs surface decorated with GMA-EDMA material [22]. And further, these substrates were applied for evacuation of PAHs via SERS detection.

In the present review, we focus on the sensor methods which are effective in the analytical evaluation of PAHs contaminated in industrial wastewater. However, this method is one of the most growing and effective methods for the new generation.

## 2. BACKGROUNDS AND COLLECTION OF PAHs

The background of PAHs is concerned with the partial combustion of organic components such as wood, coal, and oil [45]. Manufacturing of pigments, plastics, dyes, pharmaceuticals, and pesticides, as well as agrochemicals, resins, etc. are the main source of PAHs.

These are collected mainly from large point sources such as some industrial processes and incinerators. Smaller point sources are smoke from wood-burning stoves, automotive emissions, cigarettes, jet aircraft exhausts, cigar smoke, etc. Other collections of PAHs carry out sewage sludge, creosote waste, and spilling of petroleum products [46].

### 2.1 Evaluation for Recoveries of Selected Techniques

Various techniques such as fluorescence, chemiluminescence (CL), and phosphorescence are very much important. These have a wide dynamic range, are highly sensitive, have rapidity in nature, and have ease of automation. In phosphorescence, the analytical signal is evaluated by scattered light. Similarly, CL is evaluated by electromagnetic radiation through a chemical reaction. Fluorescence is widely evaluated for environmental analysis samples. Most of PAHs and other pollutants (phenol, heavy metals, etc.) are evaluated in terms of concentration up to a range of 0.01-0.03 µg/l.

Fig. 1 shows a sensor which consist of three vital systems that include a molecular recognition element (MRE), a transducer, and a detector [47]. When a target molecule of the complex sample comes in contact with the sensor, the target analyte is detected by MRE [48]. The MRE belongs to various classes, such as proteins, enzymes, and antibodies (for example enzyme-linked immunosorbent assays (ELISA); nucleic acids (ePCR to recognize RNA/DNA); aptamers (polynucleotide sequences); and carbohydrates and lectins (eLBAs) [49].

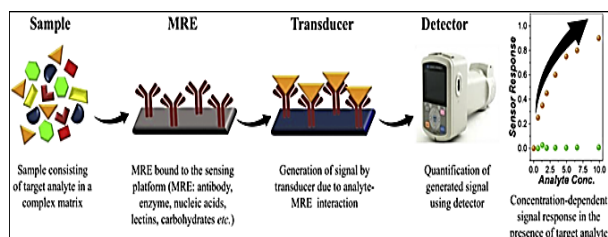


Fig. 1 – Different components of a functional sensor [57]. Copyright Elsevier (2018)

DNA-based biosensors demonstrate better detecting mechanisms along with eliminating pollutants via nanotechnology. For the detection of PAHs, a field effect transistor (FET) has been developed which has Cu<sub>2</sub>O in an appropriate quantity and DNA-doped graphene sheets (GS). FET is based on potentiometric principles which constitute a biomolecule receptor and active-surface semiconductor. The FET system (DNA/Cu<sub>2</sub>O-GS) has been designed to detect naphthalene (dynamic range of 0.2-3 µmol l<sup>-1</sup>), toluene, and benzene compounds [52].

### 3. DETERMINATION OF PAHs USING SENSOR METHOD

CL methods are usually preferred for the detection of PAHs as various constituents of PAHs have almost the same CL activity [12]. However, phosphorescence and fluorescence have advantages compared to CL as they show better response in the spectrum for minor changes. Therefore, several opto-sensors have been used for PAHs based on online pre-concentration of analytes occurred on solid supports followed by fluorescence-based measurement [53]. The analytical performance of some PAHs is shown in Table 1.

A biosensor (capacitive label-free) has been developed with the help of MIP techniques using a mono-

clonal antibody (anti-BaP). The capacitive biosensors are based on the which belongs to the impedance biosensors category. The capacitance measurement depends on their thickness layer or/and dielectric properties. It was found that MIP sensors are less sensitive than mAb-modified sensors. The mAb-modified sensors have a wide linear range, whereas for water samples, MIP sensors have high reusability [50]. From Fig. 2, the suggested fabrication method starts with electrochemical polymerization along with subsequent deposition of terthiophene-pyrene in the ratio of (4:1) prepolymerization complex onto precleaned Au QCM crystal. The removal of pyrene from the matrix through solvent extraction series which creates the electropolymerized MIP film [51].

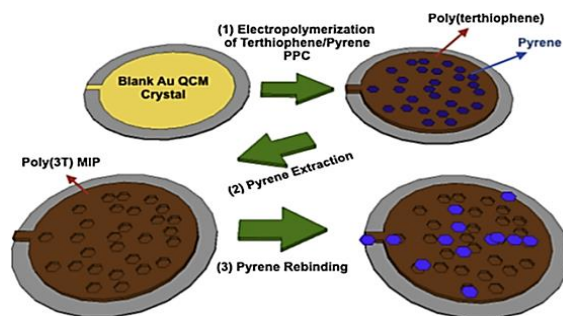


Fig. 2 – Thin film sensor fabrication diagram for pyrene-imprinted poly(terthiophene) [51]. Copyright Elsevier (2016)

### 4. PERFORMANCE EVALUATION OF THE REQUIRED QUALITY ASSURANCE IN PAH ANALYSIS

Various performance parameters such as load of detection (LOD), MIP, heavy atom induced room temperature phosphorescence (HAI-RTP), etc. have been used for quality assurance of PAHs analysis [12].

Table 1 – Analytical performance of some PAHs

PAHs	Types of water	Flow types	Comments	Ref.
BaP	Wastewater and drinking water	FIA	Online immobilization of BaP on a non-ionic resin; kex/kem: 392/406 nm; response time: 40 s	[53]
14 PAHs	River water	SLE-CF	Octadecyl membranes comprising bonded silica particles, enmeshed in a glass-fiber support; kex/kem: 330/520 nm	[54]
FLT	River water	MIP CF	Functional monomers: tetraiodobisphenol A and MDI; cross-linker: phloroglucinol; solvent: THF; template: FLT; kex/kem: 365/550,593 nm	[55]
BaP, ANT, FLT, BbF	Mineral and drinking water	FIA	PAHs were retained on Amberlite XAD 4 resin packed into the flow cell; kex/kem: 355/382 nm; response time:50 s	[53]
16 PAHs	Solution	CF	PAHs were retained on non-ionic resins packed into the flow cell	[56]

## 5. LIMITATIONS AND FUTURE DEVELOPMENTS

Since sensor methods are new developing techniques they require more reliable mechanisms and stable reaction systems for the advancement of the techniques. A more stable light source is required for monitoring the pollutants in the respective field range. This is one of the finest methods for detecting pollutants having various advantages along with some minor disadvantages.

## 6. CONCLUSIONS

Sensor methods are very important for the analytical performance of sensing methods i.e., mainly based

on flow techniques. The different components of the sensor are discussed in brief. Through MIP techniques, a capacitive label-free biosensor has been discussed using an anti-BaP monoclonal antibody. The category of a capacitive biosensor is also mentioned which is based on capacitance measurement. Biosensors along with DNA are shown in the paper. A brief discussion mentioning useful tools is also explained that is useful in removing environmental pollutants using nanotechnology. It is based on high ability and sensitivity. The field effect transistor (FET) has been illustrated.

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### Сенсорні методи виявлення поліциклічних ароматичних вуглеводнів (ПАНs) у промислових стічних водах

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Добре відомий негативний вплив промислових стічних вод на погіршення якості життя через утворення та викид небезпечних забруднюючих речовин, таких як поліциклічні ароматичні вуглеводні (ПАНs), у водойми. У статті описано різні підходи до виявлення та моніторингу ПАНs (таких як фенантрен, антрацен, флуорантен, аценафтен, хризен, пірен і перилен) у промислових стічних водах. Сенсорні методи є найбільш можливим і широко поширеним підходом для виявлення та аналізу ПАНs у промислових стічних водах. Ефективність датчика порівнюється з альтернативними методами, такими як високоефективна рідинна хроматографія (HPLC) і газова хроматографія та мас-спектрометрія (GC-MS). Відносна продуктивність різних методів виявлення на основі датчиків оцінюється в основній гарантії якості. Крім того, висвітлюються поточні обмеження та майбутні розробки методологічних підходів для їх виявлення.

**Ключові слова:** ПАНs, Промислові стічні води, Сенсорний метод, Оцінка продуктивності.