A review of the state-of-the-art wastewater quality characterization and measurement technologies. Is the shift to real-time monitoring nowadays feasible?

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ABSTRACT

Efficient characterization of wastewater stream quality is vital to ensure the safe discharge or reuse of treated wastewater (WW). There are numerous parameters employed to characterize water quality, some required by directives (e.g. biological oxygen demand (BOD), total nitrogen (TN), total phosphates (TP)), while others used for process controls (e.g. flow, temperature, pH). Well-accepted methods to assess these parameters have traditionally been laboratory-based, taking place either off-line or at-line, and presenting a significant delay between sampling and result. Alternative characterization methods can run in-line or on-line, generally being more cost-effective. Unfortunately, these methods are often not accepted when providing information to regulatory bodies. The current review aims to describe available laboratory-based approaches and compare them with innovative real-time (RT) solutions. Transitioning from laboratory-based to RT measurements means obtaining valuable process data, avoiding time delays, and the possibility to optimize the (WW) treatment management. A variety of sensor categories are examined to illustrate a general framework in which RT applications can replace longer conventional processes, with an eye toward potential drawbacks. A significant enhancement in the RT measurements can be achieved through the employment of advanced soft-sensing techniques and the Internet of Things (IoT), coupled with machine learning (ML) and artificial intelligence (AI).

1. Introduction

Health hazards and diseases caused by improperly treated wastewater (WW) are critical problems that humanity still faces today. Poor water quality may cause severe infections in humans and animals, limit food production, reduce the ecosystem's functions and biodiversity, obstruct economic growth, and result in an environmental, social, and political disaster [1]. The United Nations (UN) adopted 17 Sustainable Development Goals (SDGs) within the so-called "2030 Agenda for Sustainable Development" in 2015 [2]. The subject of the included sixth SDG refers to "Ensure availability and sustainable management of water and sanitation for all". The SDG 6 progress summary reveals that in 2020 two billion people (26% of the world's population) were lacking access to a safe drinking water source, resulting in unnecessary suffering due to water-borne diseases [3]. Furthermore, the progressing climate change and the continuous surface water pollution from contaminants of emerging concern (CECs), such as pesticides, pharmaceutics, drugs,

personal-care chemicals, and long-lasting chemicals [4], represents a grave and long-term threat to the whole of humanity and the ecosystem in which we live.

Therefore, WW quality characterization and measurement needs to be drastically improved to monitor the WW treatment and to identify pollutants of concern, ensuring its sustainable discharge in surface basins or soils. The characterization is performed by calculating specific parameters, classified into physical, chemical, and biological [5]. The physical parameters (e.g., turbidity, color, odor, solids concentration, temperature, conductivity, pH), chemical properties (e.g., chemical oxygen demand (COD), total organic carbon (TOC), TN, TP, chlorides, sulfates, alkalinity), and biological characteristics (e.g., total coliforms, Escherichia coli (E. Coli), total nematodes) have to be measured throughout the wastewater treatment plant (WWTP) to control the variability of the load [6] and the subsequent effluent quality. Discharging thresholds and sampling frequencies may vary in accordance with national or international regulations and guidelines [7].

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Nowadays, WW characterization state-of-practice relies mainly on standardized laboratory tests. These may require expensive instrumentation, specific chemical reagents, and complex, and time-consuming tests in critical environments [8]. The sample collection, preservation, and analytical procedures (including all the required reagents) are typically noted in the Standard Methods for the examination of water and wastewater [9]. Based on the investigated pollutants, the analytical experiments may be performed through titrations, spectrophotometric measurements, sensor analyses, and many other methods. Each laboratory-based analysis is unavoidably time-consuming, and results are delayed as the data must be processed [8]. Despite their widespread use, we are facing an ever-growing request for rapid procedures for the quality characterization of the WW flows. There exist real-time (RT) methods which address the time concerns, however, they are not considered entirely dependable and thus are not widely adopted yet [10].

Before proceeding, it is necessary to establish a nomenclature regarding WW monitoring. The two main monitoring methods typically employed in WWTPs are (a) traditional laboratory-based, and (b) real-time, as illustrated in Fig. 1. Within each, there are two different monitoring approaches that will be discussed.

The traditional laboratory-based methods, including off-line and at-line analyses, require identical steps from sampling to laboratory controls. However, in the off-line processes the sample is delivered to an external laboratory and not tested in the proximity of the sampling point [11]. The at-line analyses are processed near the location where the sample is collected [12]. However, building an additional area inside the WWTP may be challenging in terms of space, security (usage of chemical reagents, suitable temperature, and pressure may be required), and costs. It is possible to operate through automatic samplers, reducing the operator efforts. In any case, unavoidable delays between sample collection and data acquisition are occurring.

Switching to a RT monitoring means to obtain a processed output data in a restrained timespan after the on-field acquisition. Even though in-line (also referred to as *In situ*) or on-line (typically installed on a bypass if in-line installation is not possible) measurements have existed since the 1970s for many applications, they have been expensive and required extensive maintenance [13]. Improvements in their operation have brought about great opportunities to attain high-performing RT WW characterization. The employment of RT approaches should effectively substitute the current methods which are demanding of both time and reagents and enable rapid data availability for post-processing or remote-control uses [14]. As mentioned, the availability of reliable RT data has become essential for advanced process control in the WWTP [15]. Hence, this drives the development and optimization of sensors. Being the WW a complex mixture, sensors still need developments in order to reliably detect trace amounts of pollutants. Improved sensor capabilities allow for a more integrated control system, which in turn can lead to better management of the WW treatment plant in terms of energy and chemical reagent usage [16]. Digitalization of the WW monitoring process can be enhanced through emerging technologies, such as artificial intelligence (AI) and internet of things (IoT) [17].

A review focusing on WW online monitoring is presented by Bourgeois et al. [11]. The authors introduce standard methods and then further discuss alternative techniques including biosensors, optical sensors, sensor arrays and briefly mention soft sensors. Korostynska et al. [18] review traditionally laboratory-based and modern RT approaches in a book chapter. The most commonly measured water parameters, current techniques in monitoring wastewater quality and their limitations are presented. Furthermore, a comprehensive description regarding sensors (electromagnetic, electronic, and biosensors) for RT monitoring is reported. A review on water quality sensors is authored by Kruse [19], presenting various water quality parameters and how these can be determined, dedicating special attention to chemical sensors, mechanical, optical, and electric transduction-based sensors in a view of a reagent-free, low-maintenance and continuous monitoring application. Conventional methods and emerging technologies for general water quality monitoring were also reviewed by Ahmed et al. [20], including detection systems based on statistical inference, machine learning (ML), and IoT devices. A more recent review by Thakur and Devi [21] noted material advances, status, and future perspectives on water pollution monitoring. Further focuses on electrochemical, electrical, and optical sensors are reported. Special attention is then dedicated to IoT devices for water quality monitoring and emerging pollutants.

While the available reviews provide broad and comprehensive overviews of WW monitoring, they do not provide deep enough insight into the main characteristics of WW and practices for traditional laboratory-based methods as well as RT measurements. Moreover, they do not thoroughly explore the transition from laboratory-based tests to RT sensors in WWTP, nor do they offer an outlook of water quality characterization opportunities in a European context. A more comprehensive study focusing on these aspects is therefore needed. The present literature review aims to address these gaps while highlighting algorithm-based sensors and cutting-edge monitoring technologies.

The present review includes seven sections. The first section introduces the theme of the paper with an in-depth focus on the definition of WW monitoring, while the second describes the method for literature
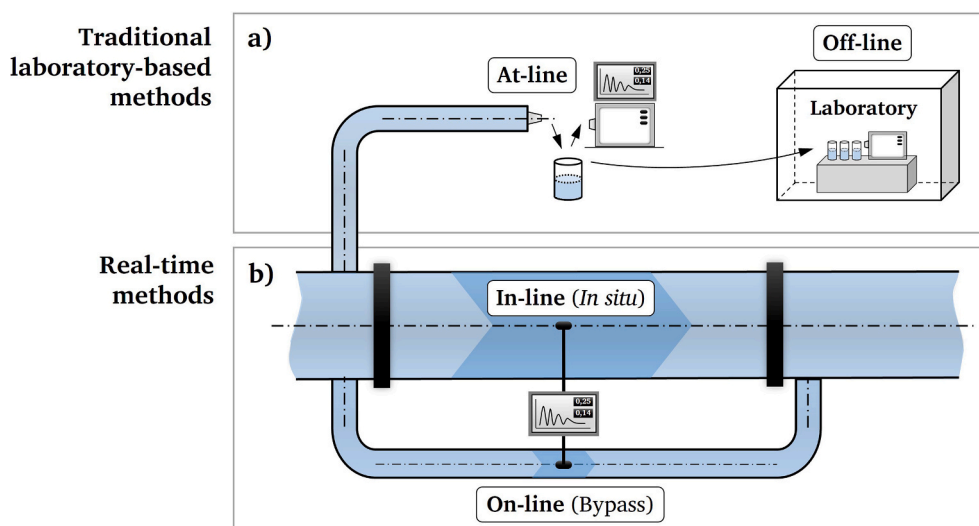

Fig. 1. Main wastewater quality characterization approaches. (a) Traditional laboratory-based and (b) real-time methods.

search and analysis. The third chapter identifies the main characteristics of WW and typical concentrations in urban WWTPs. The fourth section is dedicated to traditional laboratory-based measurements offering an extensive summary of methods, instruments, and main principles of analysis. An important passage is dedicated to the time required for each analysis to be performed and references to the new proposal concerning urban wastewater treatment. The fifth unit introduces RT measurements, summarizing parameters that are measured and sensors. The sixth section focuses on the future perspective of WW quality characterization, including emerging pollutants as well as new frontiers for RT measurements and soft sensors. The last chapter concludes the paper.

2. Literature analysis method

Publications between 2010 to 2022 have been the focus of this review. Several large scientific databases (Scopus, Web of Science Core Collection, and Dimension AI) were used to identify the topic research trend. The investigation examines the keywords "wastewater quality", inquiring about the interest in the document's title or abstract. To define the literature research trend, all types of available documents were included, i.e. research and review articles, book chapters, journal and conference papers, theses and dissertations, short reviews, encyclopedias, academic books, pre-prints, abstracts, short communications, technical reports and other scholarly literature. All the publishing access classes were considered. The total number of outputs was reported on a yearly basis and can be seen in Fig. 2. Note that the exact numbers may differ slightly based on the organizational login. The methodology to identify the research trend is comparable to several previous works [14,22,23]. The results show a consistent increase in the number of publications on the topic of WW quality. Scopus, Web of Science Core Collection, and Dimension AI give respectively a +190%, +198%, and +341% increase in publications over the period 2010-2022. Considering the field of science and engineering (S&E, all fields) from the 15 largest production countries, the mean increase in the published literature is around 52% above the same period [24]. Therefore, comparing the relative increase in the whole S&E area with the wastewater quality field, it is possible to identify the latter as attractive for researchers during recent years.

When selecting literature to be included in the current review, the aforementioned scientific databases were used in combination with Google Scholar. Documents were identified using the keywords "wastewater quality" in addition to one of the several selected knowledge-based appropriate terms, such as "biosensors", "real-time",

"characterization", "IoT", and more. Relevant guidelines, handbooks, and regulations were also included as necessary.

3. Main characteristics of wastewater

The following two categories of WW are distinct from one another: (a) domestic wastewater (DWW), and (b) non-domestic wastewater (N-DWW). DWW is defined as water altered in quality after being in contact with anthropic activities, such as human metabolism and household uses. Whereas N-DWW is discharged into collecting systems from other processes, mainly industries, exercises of trade, and institutions. The mixture of these two different wastewaters and the run-off stormwater is called urban wastewater (UWW) [25].

The composition and the load of organic compounds, solids, nutrients, micropollutants, and microorganisms may vary based on the different WW sources. Large discrepancies in properties (amount and kind of contaminants) of DWW, N-DWW, and UWW result in challenging measurements [26]. These heterogeneities may require different measurement campaign designs and equipment, adding extra cost and complexity to the characterization process. Countries and organizations commonly define specific guidelines or regulations to standardize the number of parameters (physical, chemical, and biological) needed for WW characterization. Different criteria may be required depending on the discharge location [4]. The prevailing pollutants residing in the WW are summarized in Table 1.

Commonly only a few of the parameters specified in Table 1 are mandatory water quality measurements in the treatment process. The European Council Directive 91/271/EEC defines the "Requirements for discharges from urban wastewater treatment plants" as a broad threshold or a percentage of reduction (effluent concentration compared to the influent characteristics) [32]. The WW quality is defined through an organic matter content, BOD (BOD_5 without nitrification) or COD, total suspended solids (TSS), and nutrients, through TN and TP. The discharge of WW is allowed when the analysis returns a concentration below the given limits or a reduction percentage higher than the one established by the Directive. Knowledge of the parameters at the inlet and the outlet of the WWTP is required to monitor the WW treatment progress [33]. Moreover, the European Directive suggests specific evaluation methodologies to be followed [32].

For further characterizations, COD is generally divided into its Total, Suspended, and Soluble fractions. Solids, specifically the suspended and volatile parts, are important in the treatment processes. WW streams contain concentrations of nutrients (N-P-K are the predominant ones)
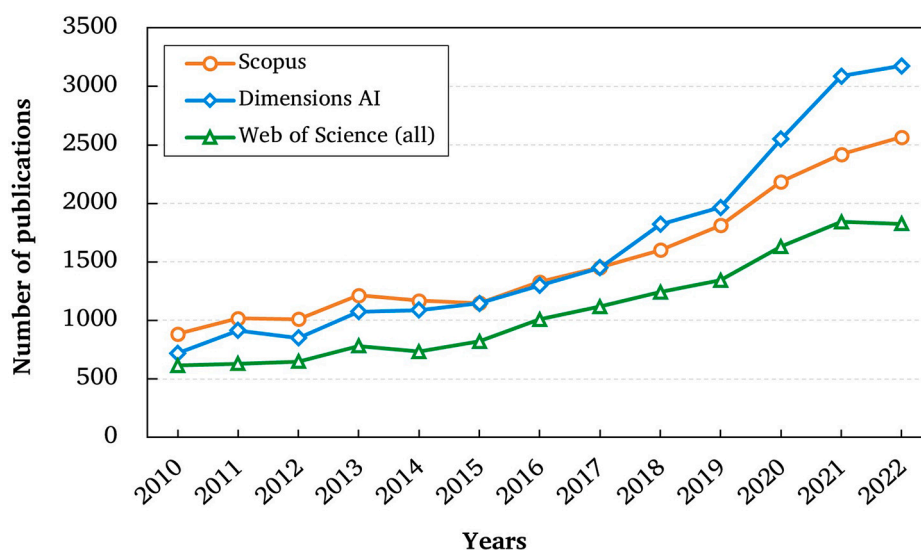

Fig. 2. Number of publications resulting from systematic web search.

Table 1
Main parameters for wastewater characterization.

Category	Parameter	Acronym	Typical concentration in urban WWTPs (mg L^{-1})*	Reference
Physico-chemical	Alkalinity	-	100-200	[5,27]
	Conductivity	-	600-800 mS cm^{-1}	[5,28]
	Dissolved oxygen	DO	5	[29]
	Ph	-	7-8	[30]
	Total hardness	-	300	[5]
	Turbidity	-	50 NTU **	[31]
Organic matter	Biochemical oxygen demand	BOD	80-300 (BOD$_5$)	[30,31]
	Chemical oxygen demand	COD	160-500 (Total) 200 (Soluble) 300 (Suspended)	[28,30]
	Total organic carbon	TOC	30-200	[29]
Solids	Total dissolved	TDS	600	[29]
	Total suspended	TSS	200-400	[28]
	Volatile suspended	VSS	320	[28]
Nutrients	Nitrogen	N	60–110 (TKN) 50-100 (NH4-N) 0.5 (Nitrate + Nitrite) 25 (Organic N)	[28,30]
	Phosphate	P	15 (TP) 10 (Ortho-P) 5 (Organic P)	[28]
	Potassium	K	9	[31]
Heavy metals	Aluminum	Al	0.6	[28]
	Cadmium	Cd	0.002	[28]
	Chromium	Cr	0.025	[28]
	Copper	Cu	0.07	[28]
	Lead	Pb	0.06	[28]
	Mercury	Hg	0.002	[28]
	Nickel	Ni	0.025	[28]
	Silver	Ag	0.007	[28]
	Zinc	Zn	0.2	[28]
Microorganisms	Coliphages	-	10^5 CFU (100 mL)$^{-1}$ ***	[28]
	Escherichia coli	E. Coli	10^7 CFU (100 mL)$^{-1}$ ***	[28]
	Roundworms	-	10 eggs L^{-1}	[28]
	Salmonella	-	150 CFU (100 mL)$^{-1}$ ***	[28]
Surfactants	Total	-	13	[31]

* Where not further specified, the concentration measure unit is mg L^{-1}
** NTU: Nephelometric Turbidity Unit
*** CFU: Colony forming unit

which vary in magnitude from 1's to 10's of mg L^{-1}. These compounds may be traceable under several forms: nitrogen as ammonia, nitrate, and nitrite; phosphorus as orthophosphates and polyphosphates, having both organic and inorganic forms. All of them are essential for nourishing numerous life forms [34]. An excessive amount of nutrients discharged in static water basins may provoke the eutrophication of the environment [35].

Nevertheless, the European Council Directive 91/271/EEC does not consider heavy metals (HMs), pathogens, and surfactants as mandatory quality controls, even though WW possesses significant amounts. HMs and microorganisms must be considered for an ecotoxicity analysis [36]. However, due to demanding analysis (mainly in terms of sample preparation and cost) less frequent HM tests are made. Microorganisms are an essential indicator due to their potential long-lasting impact on human health [37]. Moreover, surfactants are widely employed in households' activities and discharged in the drainage systems, reaching a WWTP with significant concentrations [38].

A recent proposal for a directive of the European Parliament and of the Council [25] concerning urban wastewater treatment from 26/10/2022, updated the 1991 Directive. The proposal substitutes the old thresholds and introduces relevant requirements that must be satisfied before WWTP effluent is discharged into receiving water bodies. On top of that, a quaternary treatment is requested, and "substances that can pollute water even at low concentrations" became mandatory to analyze. The latter compounds, being part of the CECs, are not given a concentration threshold but rather a minimum percentage of removal to be reached. A list of twelve compounds, divided into two groups, is reported by the new proposal. Category 1 (substances that can be very easily treated): (i) Amisulprid, (ii) Carbamazepine, (iii) Citalopram, (iv) Clarithromycin, (v) Diclofenac, (vi) Hydrochlorothiazide, (vii) Metoprolol, (viii) Venlafaxine; Category 2 (substances that can be easily disposed of): (i) Benzotriazole, (ii) Candesartan, (iii) Irbesartan, (iv) mixture of 4-Methylbenzotriazole and 6-methyl- benzotriazole. Remarkably, being these compounds currently part of the proposal, slight changes may occur when it is implemented.

Looking at a global perspective, regulations or guidelines adopted to assess the WW flows can be even stricter than EU legislation, adding a larger number of requirements or lower thresholds [7].

4. Traditional laboratory-based measurements

Several laboratory-based methods are available and recognized by the international scientific community. The most common are spectrophotometric, colorimetric, and titration measurements [39]. Sampling campaigns, obtaining appropriate containers for storage, and transportation to the laboratory are required steps preceding analysis [40]. These steps represent a non-negligible time delay toward a measurement output.

As expressed in Table 2, several standardized methods are available. By reason of well-established analytical approaches in WW laboratories, brief descriptions are given as in-depth details (such as preparation, calibration of instruments, interferences, and more) are specified in manuals and standards [9,41]. Parameters in Table 2 are categorized in alphabetical order.

The followed procedures during laboratory-based analysis are mainly reported within the Standard Methods for the Examination of Water and Wastewater [9]. Moreover, the Water Analysis Handbook from Hach [41] serves as an additional reference. The handbook contains approved methods by the United States Environmental Protection Agency (USEPA) or patented by the HACH Lange company.

As mentioned above, the prevailing parameters that must be monitored in an urban WWTP are defined by the European Council Directive 91/271/EEC [32]. In addition, the new proposal [25] introduces the TOC measure as an alternative to COD and sets the parameter TSS as optional. BOD$_5$, TOC, and COD measurement procedures are often time demanding. For instance, BOD$_5$ or ultimate BOD$_{20}$ require 5 and 20 days, respectively, to complete the analytical process [42]. A respirometric test to fractionate the COD could take more than 24 hours [43,45]; even if less time is required to attain the output values, a delay between the sampling and the availability of the result is unavoidable. Different interferences are observed inside WW when analyzed via light penetration for example TSS can inhibit adequate light transmission and reduce the accuracy of the measurements. Filtration is commonly adopted to remove settleable solids from a liquid sample. Other solid removal procedures are centrifugation or flocculation [46,47]. The standard laboratory tests for nutrient estimation, including nitrogen, phosphorus, and potassium, are mostly spectrophotometric- or colorimetric-based. Careful considerations on each test range define the requirements for sample dilution.

Other parameters, such as color, DO, turbidity, temperature, pH, alkalinity, and conductivity, are widely monitored through optical and membrane probes and can also be characterized with spectrophotometric or colorimetric methods [9]. Available tests for pathogens are

Table 2
Main traditional laboratory-based procedures for wastewater characterization.

Parameter	Method ID* or instrument	Principles and reagents	Reference
Alkalinity	2320 B. Titration	-	[9]
Biochemical oxygen demand (BOD)	5210 B. 5-Days Test	-	[9]
	5210 C. Ultimate Test	-	[9]
	5210 D. Respirometric Method	-	[9]
	Fluorescence	-	[10,42]
	UV adsorption	-	[10]
Carbon – total organic (TOC)	5310 B. High-Temperature Combustion Method	-	[9]
	5310 C. Persulfate-Ultraviolet or Heated-Persulfate Oxidation Method	-	[9]
	10128. Direct HR	Test 'N Tube Vials	[41]
	10129. Direct LR	Test 'N Tube Vials	[41]
	10173. Direct MR	Test 'N Tube Vials	[41]
	UV adsorption	-	[10]
Chemical oxygen demand (COD)	5220 B. Open reflux method	-	[9]
	5220 C. Closed reflux, titrimetric method	Oxidation, digestion, and titration	[9]
	5220 D. Closed reflux, colorimetric method	Oxidation, digestion, and spectra acquisition	[9]
	8000. Reactor digestion	-	[41]
	8000. Reactor digestion	TNT Plus	[41]
	10067. Manganese III Reactor Digestion Method (with Chloride Removal)	-	[41]
	10067. Manganese III Reactor Digestion Method (without Chloride Removal)	-	[41]
	10211. Reactor Digestion ULR TNT	-	[41]
	10212. UHR Reactor Digestion	-	[41]
	10236. Mercury-Free Reactor Digestion, TNT plus 825	-	[41]
	Respirometry	-	[43]
	UV adsorption	-	[10]
Coliforms (total, fecal, E. Coli)	8001-8001A. Most probable number (MPN)	USEPA Lauryl Tryptose Broth presumptive test with BGB, EC medium, and EC/MUG confirmation	[41]
	8091. Most probable number (MPN)	Lauryl Tryptose with MUG Broth	[41]
	8368. Most Probable Number (MPN) Method	USEPA A-1 Medium	[41]
	9221. Multiple-Tube Fermentation Technique (Total, Fecal and E. Coli)	-	[9]
	9222. Membrane Filter Technique (Total, Fecal and E. Coli)	-	[9]
	9223. Enzyme Substrate Coliform Test	-	[9]
	10018 Using LT/BCP Broth Ampules	-	[41]
Color	2120 B. Visual Comparison Method	-	[9]
	2120 C. Spectrophotometric Single-Wavelength Method	-	[9]
	2120 D. Spectrophotometric Multi-Wavelength Method	-	[9]
	2120 E. Tristimulus Spectrophotometric Method	-	[9]
	2120 F. ADMI Weighted-Ordinate Spectrophotometric Method	-	[9]
	8025. Platinum-Cobalt Standard (true and apparent)	-	[41]
Conductivity	2510 B. Laboratory Method	Conductivity cell	[9,10]
	8160. USEPA Direct measurement	Conductivity probe	[41]
	Annular ring electrode; nickel electrode; titanium or noble metal electrode	-	[10]
Dissolved oxygen (DO)	4500-O B. Iodometric Method	-	[9]
	4500-O C. Azide Modification	-	[9]
	4500-O D. Permanganate Modification	-	[9]
	4500-O E. Alum Flocculation Modification	-	[9]
	4500-O F. Copper Sulfate-Sulfamic Acid Flocculation Modification	-	[9]
	4500-O G. Membrane-Electrode Method	-	[9,10]
	4500-O H. Optical-Probe Method	-	[9]
	8157. Direct measurement	Clark-type Amperometric Sensor	[41]
	8166. HRDO	AccuVac Ampuls	[41]
	8316 Indigo Carmine	AccuVac Ampuls	[41]
	8333. Ultra High Range	AccuVac Ampuls	[41]
	10360. Direct Measurement	Luminescent dissolved oxygen (LDO) probe	[41]
	Colorimetric	Manganese sulfate and alkaline	[44]
Hardness	2340 B. By Calculation	-	[9]
	2340 C. EDTA Titrimetric Method	-	[9]
	8030. Calmagite Colorimetric Method	-	[41]
	8204. Digital Titrator EDTA (Calcium)	-	[41]
	8213. Titration Method with EDTA	Digital Titrator	[41]
	8222. Buret Method (Calcium)	-	[41]
	8226. Titration Method with EDTA	Buret Titration	[41]
	8329. Titration Method with EDTA (Sequential)	Digital Titrator	[41]
	8338. Titration Method with EDTA (Sequential)	Buret Titration	[41]
	8374. Chlorophosphonazo Rapid Liquid	Pour-Thru Cell	[41]
	Colorimetric	Calmagite	[44]

Table 2 (*continued*)

Parameter		Method ID* or instrument	Principles and reagents	Reference
Heavy metals (HMs)	Ag	8120. Colorimetric	Powder Pillows	[41]
	Al	3500-Al B. Eriochrome Cyanine R	-	[9]
		8012. Aluminon	Powder Pillows	[41]
		10215. Chromazurol S	TNTplus 848	[41]
		Colorimetric	-	[10]
		Atomic absorption spectrometry	-	[10]
	As	3500-As B. Silver Diethyldithiocarbamate	-	[9]
	Cd	8017. Dithizone	Powder Pillows	[41]
		10217. Cadion	TNTplus 852	[41]
	Cr	3500-Cr B. Colorimetric	-	[9]
		3500-Cr C. Ion Chromatographic Method	-	[9]
		8023. USEPA 1,5-Diphenylcarbohydrazide (Hexavalent)	Powder Pillows	[41]
		8024. Alkaline Hypobromite Oxidation (Total)	Powder Pillows	[41]
		10218. 1,5-Diphenylcarbohydrazide (Hexavalent)	TNTplus 854	[41]
		10219. 1,5-Diphenylcarbohydrazide (Total)	TNTplus 854	[41]
	Cu	3500-Cu B. Neocuproine Method	-	[9]
		3500-Cu B. Bathocuproine Method	-	[9]
		8026. USEPA Bicinchoninate (CuVer 2)	Powder Pillows or AccuVac Ampuls	[41]
		8143. Porphyrin	Powder Pillows	[41]
		8506. USEPA Bicinchoninate (CuVer 1)	Powder Pillows or AccuVac Ampuls	[41]
		10238. Bathocuproine	TNTplus 860	[41]
	Fe	3500-Fe B. Phenanthroline Method	-	[9]
		8008. FerroVer	Powder Pillows or Accu Vac Ampuls	[41]
		8112. TPTZ	Powder Pillows or Accu Vac Ampuls	[41]
		8146. 1,10 Phenanthroline	Powder Pillows or Accu Vac Ampuls	[41]
	Hg	10065. Cold Vapor Mercury Concentration	-	[41]
		10066. AnaLig/HgEx Mercury extraction Method	-	[41]
		Stripping	-	[44]
	Ni	8037. USEPA Heptoxime	Powder Pillows	[41]
		8150. 1-(2 Pyridylazo)-2-Napthol (PAN)	Powder Pillows	[41]
		10220. Dimethylglyoxime	TNTplus 856	[41]
	Pb	3500-Pb B. Dithizone Method	-	[9]
		8317. LeadTrak Fast Column Extraction	-	[41]
		10216. PAR	TNTplus 850	[41]
	Zi	3500-Zn B. Zincon	-	[9]
Nitrate		4500-NO$_3$ B. Ultraviolet Spectrophotometric Screening Method	-	[9]
		4500-NO$_3$ C. Second-Derivative Ultraviolet Spectrophotometric Method	-	[9]
		4500-NO$_3$ D. Nitrate Electrode Method	-	[9]
		4500-NO$_3$ E. Cadmium Reduction Method	-	[9]
		4500-NO$_3$ F. Automated Hydrazine Reduction Method	-	[9]
		4500-NO$_3$ H. Automated Cadmium Reduction Method	-	[9]
		4500-NO$_3$ I. Cadmium Reduction Flow Injection Method	-	[9]
		8358. Direct ISE	Nitrate ISE	[41]
		10020. Chromotropic Acid HR	Test 'N Tube Vials	[41]
		10206. Dimethylphenol	TNTplus 835 and TNTplus 836	[41]
		Colorimetric	Chromotropic acid	[44]
Nitrite		4500-NO$_2$ Colorimetric Method	Diazotization or Ferrous Sulfate	[9,44]
		8351. Ceric Acid Titration	Digital Titrator	[41]
		8507. Diazotization Chromotropic Acid	Test 'N Tube Vials	[41]
		10019. Diazotization	Test 'N Tube Vials	[41]
		10207. USEPA Diazotization	TNTplus 839	[41]
		10237. USEPA Diazotization	TNTplus 840	[41]
Nitrogen	Ammonia	4500-NH$_3$ C. Titrimetric Method	-	[9]
		4500-NH$_3$ D. Ammonia-Selective Electrode Method	-	[9]
		4500-NH$_3$ E. Ammonia-Selective Electrode Method Using Known Addition	-	[9]
		4500-NH$_3$ F. Phenate Method	-	[9]
		4500-NH$_3$ G. Automated Phenate Method	-	[9]
		4500-NH$_3$ H. Flow Injection Analysis	-	[9]
		8038. USEPA Nessler	Reagent Solution	[41]
		8155. Salicylate	Powder Pillows	[41]
		10023. Salicylate	Test 'N Tube Vials	[41]
		10031. Salicylate	Test 'N Tube Vials	[41]
		10201. Indophenol	Powder Pillows	[41]
		10205. Salicylate	TNTplus 830 (ULR) – 831 (LR) – 832 (HR)	[41]
		Colorimetric	Indophenol	[10]
			Berthelot reaction	[10]
		Ion chromatography	-	[10]
	Total (TN)	4500-N$_{org}$ B. Macro-Kjeldahl Method	-	[9]
		4500-N$_{org}$ C. Semi-Micro-Kjeldahl Method	-	[9]
		4500-N B. Uv/Persulfate Digestion and Oxidation	-	[9]
		4500-N C. Persulfate Method	-	[9]
		4500-N D. Conductimetric Determination (Inorganic N)	-	[9]
		8075. Nessler with digestion (Kjeldahl)	Nessler reagent	[41]

Table 2 (*continued*)

Parameter		Method ID* or instrument	Principles and reagents	Reference
pH		10021. Titanium Trichloride Reduction Colorimetric	Test 'N Tube Vials	[41]
			Chromotropic acid	[44]
		4500-H$^+$ B. Electrometric Method	-	[9]
		Colorimetric	Litmus (pH 5–8) methyl orange 2–7.0 mmol L^{-1} H+ (pH 3.1–4.4)	[44]
		Ion-sensitive field effect transistor (ISFET)	-	[10]
		Titration	Sodium hydroxide	[10]
Phosphates	Ortho	4500-P G. Flow Injection Analysis	-	[9]
		8048. USEPA PhosVer 3	Test 'N Tube Vials	[41]
		8048. USEPA PhosVer 3 (Ascorbic Acid)	Powder Pillows or AccuVac Ampuls	[41]
		8114. Molybdovanadate	Test 'N Tube Vials or Reagent Solution	[41]
		8178. Amino Acid	Reagent Solution	[41]
		10055. Ascorbic Acid Rapid Liquid Method	-	[41]
		10209. Ascorbic Acid	TNTplus 843 (LR) – 844(HR) – 845 (UHR)	[41]
		10214. Molybdovanadate	TNTplus 846	[41]
	Total (TP)	4500-P C. Vanadomolybdophosphoric Acid Colorimetric Method	-	[9]
		4500-P D. Stannous Chloride Method	-	[9]
		4500-P E. Ascorbic Acid Method	-	[9]
		4500-P F. Automated Ascorbic Acid Reduction Method	-	[9]
		4500-P H. Manual Digestion and Flow Injection Analysis	-	[9]
		4500-P I. UV/Persulfate Digestion and Flow Injection Analysis	-	[9]
		4500-P J. Persulfate Method	-	[9]
Potassium (K)		3500-K B. Flame Photometric Method	-	[9]
		3500-K C. Potassium-Selective Electrode Method	-	[9]
		8049. Tetraphenylborate	Powder Pillows	[41]
Salinity		2520 B. Electrical Conductivity Method	-	[9]
		2520 C. Density Method	-	[9]
		2520 D. Algorithm of Practical Salinity	-	[9]
		10073. Titration	Mercuric Nitrite	[41]
Solids	Total (TS)	2540 B. Dried at 103-105°C	-	[9]
		8271. USEPA Gravimetric	-	[41]
	Total dissolved (TDS)	2540 C. Dried at 180°C	-	[9]
		8163. USEPA Gravimetric	-	[41]
	Total suspended (TSS)	2540 D. Dried at 103-105°C	-	[9]
		Centrifuging and drying	-	[32]
		8006. Photometric	-	[41]
	Volatile suspended (VSS)	2540 E. Ignited at 550°C	-	[9]
		2540 G.	-	[9]
		8164. USEPA Gravimetric	-	[41]
Surfactants		5540 B. Separation by Sublation	-	[9]
		5540 C. Anionic Surfactants as MBAS	Colorimetric equipment	[9]
		5540 D. Nonionic Surfactants as CTAS	Ion-exchange column	[9]
		8028. Cristal violet (anionic)	-	[41]
Temperature		2550 B. Laboratory and Field Methods	-	[9]
		Thermistor	-	[10]
Turbidity		2130 B. Nephelometric Method	-	[9,10]

* Methods reported with the number of references and name

mostly related to the most probable number (MPN) and membrane filtration. These processes are characterized by a long preparation and incubation of the cultures [48]. The Table 2 parameters not covered by the directive, such as surfactants and HMs, can be assessed with a vast range of laboratory instruments. Almost all laboratory-based analyses are time intensive, from the sample preparation (i.e. filtration, dilution) to the duration of the laboratory analysis (i.e. digestion, reaction time, mixing) [41]. Table 3 reports the mean duration of the overall laboratory test, maintaining the parameters defined in Table 2.

It is important to clarify that these durations must be considered as the time needed to perform the test and read the result on the device, starting with a sample fully prepared. The sample collection (normally 24 hours), transport to the laboratory location (variable from minutes to hours), and preparation (variable from minutes to hours) are not included. Moreover, the reagents preparation and the test management may augment the required time.

In addition to the aforementioned methods, high-precision instruments (tracing parts per billion (ppb) concentrations) may be employed to characterize the liquid matrix [50]. However, longer sample preparations, expensive reagents, and higher process complexity

are possible drawbacks. Instruments, such as Inductively coupled plasma-optical emission spectrometry/mass spectrometry (ICP-OES/MS), nuclear magnetic resonance (NMR), and high-performance liquid chromatography (HPLC), are typically used for qualitative assessments [51–53]. However, the requirements and costs for installation, standard references, and maintenance requirements are onerous. The ICP-OES/MS principle is based on a coupled effect between inductively coupled plasma (ICP), optical emission spectrometry (OES), (sometimes also referred to as atomic emission spectrometry (AES)) and mass spectroscopy (MS). This instrument can determine the concentration of metallic and non-metallic inorganic substances at ppb [54]. Gas chromatography (GC) is used for the characterization of several WW micropollutants [55]. Moreover, fluorescence techniques are employed for WWTP process control [42,56]. Laboratory analyses along with nuclear magnetic resonance (NMR) are useful to detect liquid organic contents. A wide variety of compounds may successfully be quantified through the ^{1}H NMR [57]. Furthermore, solid-state ^{13}C NMR revealed promising features for its application as a qualitative detection of organic matter [58]. Evaluations of several HMs have been performed through flame atomic absorption spectrophotometry (FAAS) on water samples [20,59,60].

Table 3
Relation between the compound measured in the laboratory and the duration of the analysis.

Parameter		Duration	Reference
Alkalinity		< 1 min *	[9]
Biochemical oxygen demand (BOD)		5-20 d **	[10,41,42]
Carbon – total organic (TOC)		2 h	[9,10,41,49]
Chemical oxygen demand (COD)		> 2 h	[9,10,41,43]
Coliforms (total, fecal, E. Coli)		> 24 h	[9,41]
Color		< 1 min *	[9,41]
Conductivity		< 1 min *	[9,10,41]
Dissolved oxygen (DO)		< 1 min *	[9,10,41,44]
Hardness		< 1 min *	[9,41,44]
Heavy metals (HMs)	Ag	> 2 min	[41]
	Al	> 5-15 min **	[9,10,41]
	As	> 45 min	[9]
	Cd	> 2 min	[41]
	Cr	> 10 min	[9,41]
	Cu	> 2 min	[9,41]
	Fe	> 3 min	[9,41]
	Hg	> 2 h	[41,44]
	Ni	> 15 min	[41]
	Pb	> 1 min	[9,41]
	Zi	> 3 min	[9]
Nitrate		> 5 min	[9,41,44]
Nitrite		> 20 min	[9,41,44]
Nitrogen	Ammonia	> 1-20 min **	[9,10,41]
	Total (TN)	> 2-40 min **	[9,41,44]
pH		< 1 min *	[10,41,44]
Phosphates	Ortho	> 2 min	[9,41]
	Total (TP)	> 2 min	[9]
Potassium (K)		> 3 min	[9,41]
Salinity		< 1 min *	[9,41]
Solids	Total (TS)	> 6 h	[9,41]
	Total dissolved (TDS)	> 2 h	[9,41]
	Total suspended (TSS)	> 2 h	[9,41]
	Volatile suspended (VSS)	> 2-7 h **	[9,41]
Surfactants		> 30 min	[9,41]
Temperature		< 1 min *	[9,10]
Turbidity		< 1 min *	[9,10]

* Measured with digital instruments, outdated laboratory tests for that parameter.
** Depending on the adopted method.

Towards the assessment of the twelve emerging compounds from the 2022 proposal [25], a brief investigation is reported. The following references are obtained by searching in the above-mentioned databases for the name of the substance and "wastewater". The most relevant results were examined and reported in Table 4. It is common that no

standard laboratory tests (i.e. titration, colorimetric evaluations) are available for their measurement except advanced and demanding instruments, such as liquid chromatography (LC), HPLC, ultra HPLC (UHPLC), reversed-phase LC (RPLC), diode-array detection (DAD), and GC, regularly coupled with MS.

The identification of a single compound inside WW can be challenging due to the large number and variety of compounds present and potential interferences. The availability of a reference standard for the compound of interest can impact the applicability of the analytical methodologies, such as LC which is not suitable to identify a single target without a reference [86]. However, analytical procedures that do not require standard references for target identification are potential solutions (non-target screening) [87]. The concentrations of micrograms per liter or lower are currently challenging to be measured. Regardless of the difficulties, the monitoring of WWTP discharges into water bodies must consider these CECs. Removal through tertiary [88] or quaternary [25] treatments is required to meet new regulatory requirements.

5. Overview of real-time measurements

RT techniques may provide a solution to overcome struggles regarding the preparation, collection, and storage of WW samples, resulting in faster data acquisition from WWTP flows [89]. The RT measurements, as previously mentioned, are carried out with sensor-based in-line or on-line monitoring. Precise, accurate, cost-effective, operator-friendly, and fast-response procedures must be introduced to optimize the WWTP and improve control and the detection of abnormalities throughout the processes [15]. Among all the above-mentioned parameters, WW inflow concentrations of BOD, COD, TN, and TP and DO values throughout the treatment are most influential to the performance of WWTPs [90]. By characterizing the inflow, aeration processes and chemical addition can be optimized in RT [91,92]. Focusing on the energy and the operational costs, several literature studies reported monitoring strategies for process control. Continuous influent RT characterization of pilot- and full-scale plants are shown to be capable of reducing the annual energy consumption from 13 to 30%, through improving the accuracy of process control [15]. So, developed sensors may improve WWTP supervision, leading to an optimal management and overview of the WWTP's energy consumption [93]. Overall, it appears that implementing RT measurements of relevant parameters can improve the operation of the WWTP [94]. However, as with most technologies, improper use can cause malfunctions which represents a grave drawback [93]. The sensor installation and maintenance need to be considered; periodic calibration and repairs could be an inconvenience for the WWTP administration [95,96]. Furthermore, a robust elaboration/processing model is needed to set the variables and manage

Table 4
Substances that can pollute water at low concentrations.

Substance	CAS number	Analytical method	Usage	Mean UWW concentration (mg L^{-1})	Reference
Amisulprid	71675-85-9	UHPLC-MS, LC-HRMS	Atypical antipsychotics	10^{-6}-10^{-7}	[61]
Carbamazepine	298-46-4	LC-MS/MS, GC-MS/MS, HPLC-MS	Analgesic, antiepileptic	10^{-6}-10^{-8}	[62–65]
Citalopram	59729-33-8	LC-MS/MS, HPLC-DAD	Antidepressant	10^{-7}	[66,67]
Clarithromycin	81103-11-9	LC-MS/MS; HPLC-MS/MS	Antibiotic	10^{-6}	[68,69]
Diclofenac	15307-86-5	LC-MS/MS, GC-MS	Analgesic, anti-inflammatory	10^{-6}-10^{-8}	[65,70,71]
Hydrochlorothiazide	58-93-5	LC-MS/MS	Antihypertensive	10^{-6}	[72]
Metoprolol	37350-58-6	LC-MS/MS	Antihypertensive	10^{-7} -10^{-8}	[73]
Venlafaxine	93413-69-5	HPLC-MS; GC-MS	Antidepressant	10^{-6}-10^{-8}	[74–77]
Benzotriazole	95-14-7	LC-MS/MS; HPLC-MS	Corrosion inhibitor	10^{-5}-10^{-8}	[78–81]
Candesartan	139481-59-7	RPLC-MS; LC-MS	Antihypertensive	10^{-6}-10^{-7}	[82,83]
Irbesartan	138402-11-6	RPLC-MS; HPLC-MS; LC-MS	Antihypertensive	10^{-6}-10^{-7}	[82–84]
Mixture of 4-Methylbenzotriazole and 6-methyl-benzotriazole	29878-31-7 and 136-85-6	LC–MS/MS; HPLC-MS/MS	Corrosion inhibitors	10^{-6}	[79,85]

the extensive datasets acquired during the continuous measurement [89]. The main RT available technologies are reported in Table. Parameters are listed in alphabetical order.

Available sensors allow the measurements of a wide range of parameters, useful for characterizing WW. Each parameter reported in Table 5 is related to an appropriate sensor for its evaluation. Principles of measurement are also reported. Innovative applications consist of fiber optics, potentiometric, and electrochemical biosensors, adopted in the *In situ* WW quality assessment [103,108].

In general, a sensor is responsive to changes of certain liquid properties. The acquired signal is transduced into a readable value [109,110]. Input sources may vary from sensor to sensor: voltage, current, conductivity, and dielectric constant are monitorable.

5.1. Optical sensors

Optical sensors are referred to when a direct optical property of WW can be measured [111] differencing from chemical-optical sensors where an analyte-sensitive receptor is required [112,113]. Optical ultraviolet-visible (UV-Vis) sensors are widely employed; however, issues during *In situ* analysis such as sensor fouling (i.e. various deposit accumulation on the optical surfaces) often occur [98]. Among all the traceable WW compounds, only a fraction of them absorb light in the UV-Vis range (wavelength from 190 to 850 nm) [114]. This results in a need to combine UV-Vis sensors with other optical analytical methods in order to cover a wider wavelength spectrum. Infrared (IR), fluorescence, and Raman spectrophotometric investigations are nowadays applied in WWTP RT monitoring [10]. Despite technological developments with the achievement of higher precisions, optical methods still in many cases remain bound to laboratory validation and the reusability of these sensors has still to be assessed [107]. Promising *In situ* analysis can be achieved by optical fiber sensors that are easy to place in hard-to-reach areas due to small probe size and are resilient in tough conditions (i.e. noise, vibrations, and heat) [115]. The composition of optical fiber sensors differs from the electrical due to the introduction of glass fibers instead of copper cables, capturing light as an indicator. The fiber's protective coating plays a significant role in avoiding external interferences [116]. In addition, transducers and detectors are required [98]. Polymeric fibers are also used in these sensors. Nowadays, commercially available fiber optic sensors may cover the whole UV-Vis-IR spectrum. Detection limit improvements are under development, with novel materials implementation [108].

5.2. Chemical sensors

Chemical sensors are able to detect particular chemical properties from an analyte. The detected input has to be further transduced into a readable physical signal. Three main mechanisms of transduction are available: (a) mechanical, (b) optical, and (c) electrical [19]. To elaborate on these: (a) An atom, composing a molecule, fills a precise amount of physical space (i.e. steric effect concept). Mechanical transduction works through the understanding of this physical space taken by an analyte species. Therefore, the signal, converted before the final recognition, is uniquely defined. Previous studies employed mechanical sensors for the evaluation of ammonium ions [117] and total hardness [118]. Continuing to (b), optical transduction identifies changes in the electronic structure of the receptor molecule. An analyte detector is required to provide acquirable optical signals [113,119]. Absorbance, chemiluminescence, fluorescence, and phosphorescence are evaluated as optical properties [15]. Optical fibers are adopted as transductor as well [120]. As reported in Table, chemical sensors with optical transductor may identify several parameters, such as pH, total hardness, nitrates, phosphates, different metal cations, DO, and surfactants [19]. Finally, (c), the electrical transductor requires direct contact with the liquid sample. The presence of high solid concentrations or aggressive compounds may lead to malfunctions in sensors of this type. The

Table 5

Available real-time measurement processes for wastewater characterization.

Parameter		Instrument	Principle	Reference
Alkalinity		Chemical sensor	Optical, electrical	[19]
Biochemical oxygen demand (BOD)		Microbial fuel cell		[97]
		Fiber optic sensor		[98]
Carbon – total organic (TOC)		Optical chemical sensor	Infrared absorption	[19]
Chemical oxygen demand (COD)		Microbial fuel cell		[97]
		Fiber optic sensor		[98]
		Electromagnetic wave sensor		[99]
Color		Fiber optic sensor		[98]
Conductivity		Conductometric sensor		[100]
Dissolved oxygen (DO)		Electrochemical sensor	Amperometric, Potentiometric	[19,101]
		Optical chemical sensor	Luminescence, fluorescence	[19]
		Microbial fuel cell		[97]
Escherichia coli (E. Coli)		Biosensors	Optical Amperometric	[23,102]
		Electromagnetic wave sensor		[99]
Hardness		Chemical sensor	Mechanical	[19]
		Electrochemical sensor	Potentiometric	[19]
		Optical chemical sensor	Colorimetric, fluorescence	[19]
Heavy metals (HMs)	Ag	Biosensor	Colorimetric	[103]
	Al	Optical chemical sensor	Fluorescence	[19]
	Cd	Optical chemical sensor	Colorimetric, fluorescence	[19]
		Electrochemical sensor	Anodic stripping voltammetry	[19]
		Biosensor		[103]
	Cr	Optical chemical sensor	Fluorescence	[19]
		Biosensor	Electrobiochemical	[103]
	Cu	Optical chemical sensor	Colorimetric, fluorescence	[19]
		Biosensor	Fluorescence	[103]
	Hg	Optical chemical sensor	Absorption, fluorescence, colorimetric	[19]
		Electrochemical sensor	Chemiresistive	[19]
		Biosensor	Bioluminescence, Optical, Piezoelectric	[103]
	Ni	Optical chemical sensor	Colorimetric	[19,104]
	Pb	Electrochemical sensor		[19]
		Optical chemical sensor	Colorimetric	[19]
		Biosensor	Electrochemical	[103]
	Zn	Electrochemical sensor	Anodic stripping voltammetry	[19]
Nitrate		Electrochemical sensor	Potentiometric, amperometric, voltametric	[19]
		Optical chemical sensor	Colorimetric, fluorescence	[19]
Nitrite		Optical chemical sensor	Absorbance, fluorescence	[19]
		Electrochemical sensor	Potentiometric	[19]
		Electromagnetic wave sensor		[99]
Nitrogen	Ammonia Total (TN)	Chemical sensor	Mechanical	[19]
		Ion-selective membranes sensor		[105]
pH		Optical chemical sensor	Raman, fluorescence	[19]

Table 5 (*continued*)

Parameter	Instrument	Principle	Reference
	Electrochemical sensor	Potentiometric	[19,106]
	Electrical sensor	Chemiresistive	[19]
Phosphates	Electrochemical sensor	Potentiometric, amperometric	[19]
	Optical chemical sensor	Fluorescence	[19]
Potassium (K)	Biosensors	Potentiometry	[106]
Salmonella spp.	Biosensors	Optical Amperometric Conductometric	[102]
Solids, total suspended (TSS)	Optical chemical sensor		[107]
Surfactants	Electrochemical sensor	Potentiometric, conductometric	[19]

principle of detection is based mainly on potentiometric, amperometric, voltametric, and electrochemical impedance measurements [19].

Recent studies have revealed that the chemical sensors may be applied successfully for RT *In situ* analyses [14,100]. Further studies are needed to evaluate their long-term applicability, overtaking additional WW pre-treatments to enhance the signal's acquisition [121].

5.3. Biosensors

Biosensors are currently employed for the evaluation of organic compounds, microorganisms, and heavy metals [103]. Biosensors are made up of two main components: a bioreceptor and a transducer [122]. Amperometric biosensors are used for BOD and COD assessments [123]. Heat variations are also considered in COD measurements [124]. Persistent organic compounds are traceable in WWTP connected to industrial discharges [125]. Microbial fuel cell (MFC)-based biosensors have been introduced in the detection of organic compounds [97,126]. The application of biosensing techniques may also provide RT controls for microorganisms [127]. European guidelines and regulations for the assessment of Total Coliforms, E. Coli, Enterococcus, and Intestinal Nematodes as microorganism indicators [7] have conferred even more importance to their RT surveillance [102]. Recent studies focused on the detection of microorganisms in WW through optical and electrochemical biosensors. Surface plasmon resonance (SPR) [128,129], resonant mirror (RM) [106], sensitive chemiluminescence immunosensors [130], and amperometric biosensors [131,132] were reviewed, resulting in precisions closer to μg L^{-1}. Increasing attention is also given to the assessment of HMs in waste liquids [133]. Biosensors and nano-biosensors have been introduced in WW analysis as a substitute for analytical methods. Even though laboratory analysis possesses sufficient precision and reliability, the establishment of sensor-based *In situ* measurements is preferred due to the faster response time, as previously mentioned. Taking advantage of bacteria as a bioluminescence source, microbial biosensors are able to detect the presence of low-concentrated HMs [104,134]. Nano-biosensors, based on the fluorescence resonance energy transfer (FRET) principle, can measure trace concentrations of metals. A broad range of enzymes have been applied in these RT monitoring techniques through enzyme-based electrochemical biosensors and aptasensors [135,136], with the potential addition of nanomaterials to enhance their detectable capabilities [137]. Moreover, ion-selective polymer membranes [138,139] and fluorescent-based microfluidic devices [140–142] have shown potential in pollution control of aqueous solutions. A combination of two or more methods [143] or materials [144] may be convenient. Recent studies have reported that MFC-based biosensors have the potential to be a cost-effective device for WW RT monitoring [23,97]. Small-scale devices were developed even though their stability, sensitivity, repeatability, and selectivity have still to be validated. MFCs have been additionally tested for the investigation

of BOD, COD, and DO [145–147].

5.4. Ion-selective membrane sensors

Ion-selective membrane (ISM) sensors are established detectors for measuring the ammonium concentration in water [148]. A new generation of solid-state ion-selective membranes (S-ISMs) is studied to overtake the high costs and fragility of the available ISMs [149]. S-ISM performances have shown to be more durable, accurate, have a more rapid response time, and be cheaper than ISM sensors [105]. Moreover, electrospray printing technology was successfully employed in the preparation of an ultrathin S-ISM. Results showed an increase in the sensor response, still maintaining a restrained error [149]. Metal ions are also monitored in RT within industrial WW flows [150,151] and WWTPs [152] using these technologies.

5.5. Lab-on-chip and electrochemical sensor-based systems

Lab-on-chip (LOC) and electrochemical sensor-based systems were tested for WW characterization [153]. The introduction of *In situ* LOC aimed to transform the whole analytical laboratory process (sampling, preparation, addition of reagents, chemical reactions, and measurements) into a simplified, fast-responding, and restrained-volume approach. The integration of microelectrodes and microelectromechanical systems with potentiometric and voltametric sensors was assessed. Applications on real plants for nutrients evaluation are reported in the literature [154,155].

5.6. Electromagnetic wave sensors

Recent applications of electromagnetic wave (EM-W) sensors aimed to control WW pollution through important parameters like COD and nitrates [156], HMs [157], E. coli, and Pseudomonas Aeruginosa [99] with frequencies in the range of 0-15 GHz. In order to evaluate Cu, Ni, and Zn concentrations, the EM-W reflection technique was flanked with an open-ended coaxial sensor. Moreover, phosphates, ammonium, and other HMs (Hg, Pb, Cr) were tested at 1-10 GHz [158]. The electromagnetic waves, interacting directly with the analyte, generate a singular response that is specifically related to the WW properties [159]. Microwave sensing allows the non-invasive RT characterization of WW. Considering variations between the output transmittance (or reflectance) wave and the input signals at peculiar frequencies, the composition of the liquid can be established. The fluctuations of the surrounding environmental conditions (temperature, pH, turbidity, etc.) have to be taken into account while comparing analyses along time [160,161].

5.7. Electronic tongues

An electronic tongue (ET) structure is based on a group of low selective sensors that, through the avail of data processing analysis, emerged to be suitable for a quantitative WW characterization [162]. The voltametric ET is the most diffused detector, assessing the potential variations over time. These devices are composed of a set of metallic sensors covered with films (useful for sensitivity improvement) coupled with a reference electrode [163]. ETs are efficient, low-volume requiring, cost-effective, faster-response, and automatic detectors. Moreover, the *In situ* measurements do not require qualified operators [164]. Amperometric bioelectronic tongues are likewise considered in literature [165]. principal component analysis (PCA), partial least square analysis (PLS) and neural networks analysis (NNA) are diffused elaboration processes to handle the huge quantity of output acquisitions [15].

5.8. Final remarks

The use of hardware redundancy, multiple sensors for the same parameter placed at a single measurement location, is a common way to detect errors in the measurements provided by RT sensors. Another approach is the use of sensor fusion technology, which leads to an improved ability to detect errors through combining information from multiple measurement types [130]. Sensor fusion can also take the form of analytical redundancy, as opposed to hardware redundancy, through the use of different models, soft sensors, or analyses of the measurements [166]. Despite having a single sensor, more signals could highlight a malfunction, or error, in the measurements [167]. Similarly, data reconciliation techniques can be employed to correct, validate, and harmonize data from disparate sensor sources, enhancing accuracy and reliability in the detection of discrepancies or errors [168]. Several devices are being, and have been, developed for RT measurements of WW quality parameters. However, a small percentage of them are suitable for multiple purposes. Sensors are ordinarily built for a specific purpose and are not reusable in other environments [21]. Several challenges are faced during RT analysis, exceeding precision and general applicability. The harsh conditions in WW flows require sensors either specifically designed to withstand the hostile environment or extensive maintenance [169]. Both cases are costly and time-consuming. Fault detection is likewise essential. For instance, air bubble interference may alter the optical sensor outputs, and clogging due to the presence of small particulates can cause permanent damage [170]. Moreover, biofouling (an undesired formation of bacteria colonies that alters the sensor performance [171]) is a challenge for any sensor in direct liquid contact. Copper-based and plastic protections are valid anti-biofouling countermeasures [19]. Thus, the sensors' lifetime and performance in WW are strongly impacted by these malfunctions. As briefly mentioned earlier, the need for correct installation, maintenance, and continuous calibration of these RT sensors is therefore essential for ensuring measurements of a good and reliable quality. The question of when to calibrate a sensor is of great importance, and, when laboratory measurements are used for calibration of RT sensors, an awareness of the uncertainties introduced is essential [172]. Data validation and fault detection methods should be considered and prioritized when working with RT measurements. For example, monitoring of measurement ranges, variance analysis, and well-known control chart techniques are commonly used tools for validating the quality of the signal [173].

Notwithstanding, it is still possible to notice a dearth in RT monitoring towards the contaminants of emerging concerns. The 2022 proposal forces the WWTP stakeholders to control the removal rate of twelve emerging pharmaceutical parameters. Nowadays, exclusively the non-targeted advanced laboratory analysis can determine their concentration [67]. Moreover, WW flows may contain microplastics, personal care products, pesticides, and drugs [174,175] which are not required to be monitored but can be harmful to the surrounding environment. Thus, WW flows are one of the hardest liquid solutions to deal with, especially due to the heavy load of hazardous micropollutants. In-line sensor-based processes are not recognized as reliably able to monitor trace elements at low concentrations (below a ppm). So, WW outflows could not be completely characterized in RT as soon as all the trace contaminants are. Further studies need to be designed to give solutions for their employment in real WWTPs. Furthermore, high-sensitivity sensors are required to reach advanced precision, moving closer to the $\mu g\,L^{-1}$ thresholds and beyond.

6. Future prospectives of wastewater quality characterization

As the requirements placed on WWTP discharges become stricter, evidenced by changes to the urban WW treatment directives (for instance, Council Directive 91/271/EEC required a 80% reduction in TP and minimum 70% reduction in TN [32]; these will change to 90% and 85% reductions respectively considering the recent proposal for a directive of the European Parliament and of the Council concerning urban wastewater treatment 26/10/2022 [25]), some adjustments in the WWTP have to be considered. While these changes may involve infrastructural modifications, a more cost-effective first approach would develop the monitoring and control of the process. Various studies over the years have shown that significant improvements on nutrient removal and process efficiency can be made simply through the use of advanced process control [13,176], and as the available sensor and measurement technology for water quality characterization improves so do the options and opportunities for the implementation of control.

Another important aspect of the tightening of regulations is the increase in monitoring requirements described in the directives that has been discussed in previous sections. Currently, in order to show compliance with regulatory limits, parameters must be monitored through reference measurement methods which are often time-consuming and expensive (Section 4). A question that should be pursued by the research community is: *How can the RT measurement become reliable enough to be included in directives and regulations and carry some of the regulatory monitoring burden?* This will require extreme improvements in trust of the technology, as well as methods for proving its trustworthiness. However, this is immensely important for the field of RT WW quality characterization – while not all treatment plants may be interested in the potential of improved monitoring capabilities with regards to control opportunities, all treatment plants must be concerned monitoring with regards to regulations. Advancing technology to enhance efficiency and reduce costs in this domain is of significant importance.

While the focus of the discussion thus far has been on the treatment of WW, additional parameters must be characterized to support water reuse [177] due to increased water scarcity and deterioration of water quality caused by climate change, increasing unpredictable weather events and drought [178]. In line with this, the restrictions for reuse of water have been defined (Regulation (EU) 2020/741 [179] on minimum requirements for water reuse). These reuse restrictions detail quality parameters such as E. coli, BOD_5, TSS, Turbidity and Legionella spp. which must be monitored with specifically defined frequencies. There are also requirements to perform risk assessments, possibly identifying additional or stricter limitations; including HMs, pesticides, disinfection by-products, pharmaceuticals, and micro-plastics. A special attention must be dedicated to antibiotic resistance genes (ARGs), antibiotic resistant bacteria (ARBs) and per- and polyfluoroalkyl substances (PFAS), which are toxic, persistent, and bio-accumulative chemicals [180]. This introduces yet another driving force behind the improvement and development of RT water quality characterization methods, for possibly an even wider range of quality parameters than those required by urban WW treatment regulations.

Aside from regulatory push factors, there are many technological factors that are currently impacting the research progress in these areas. Readily available computing power, the sudden improvements to AI and ML and their accessibility, as well as the quality of cheap and readily available IoT sensors, to name a few, are major contributors to the direction that research in this field will likely take over the coming years. Many of these technologies are not new, but the prevalence of their use and their general acceptance is expected to improve greatly.

6.1. Soft sensors and IoTs

Soft sensors have emerged as one of the critical technologies for monitoring and controlling WW treatment processes in RT, thanks to advances in data analytics, digitalization, and increased availability of computational power. Soft sensors are developed by using data from multiple hardware sensors to create a mathematical model that outputs a target parameter estimation (Fig. 3).

The status and advances of soft sensors are extensively discussed in several comprehensive studies, such as Haimi et al. [181], that focus on advancing biological WW treatment through data-driven soft sensors,
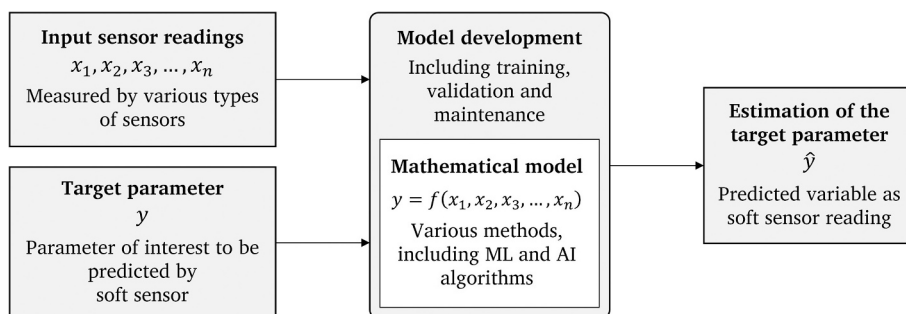
Fig. 3. Soft sensor concept overview, adapted from [94].

emphasizing their utility in RT monitoring and optimization. The review summarizes mathematical methods, processes where soft sensors are employed, if the soft sensors are practically implemented, and presents a list of estimated target parameters. Special attention is dedicated to process monitoring and fault detection. A more recent review by Ching et al. [94] extensively examines soft sensor development methods and their effectiveness in relation to hardware technology. The authors list input sensors used for soft sensor development, mathematical methods as well as target parameters and resulting model performances. Furthermore, artificial neural network (ANN) applications for soft-sensing and comparison of different architectures are reviewed by Wang et al. [182]. The study outlines challenges in soft-sensing model applications, including low data effectiveness (high complexity and external disturbances of WWTP) and the need for robust, efficient, and accurate models as well as the importance of reliability and uncertainty analyses.

Based on current literature, the input sensors (i.e. predictors) utilized for development of soft sensors typically include the following parameters, ranging from influent to effluent: TSS [183–185], COD [185–187], TP [183,187], TN [183,186], ammonia [185,188], oxidation-reduction potential (ORP) [187,189], DO [187,190], flowrate [186,191], pH [184,185,192], temperature [184,185], BOD [183,184,187], nitrite and nitrate [185,188], filling time [193], reaction time [193], aeration rate [193,194], recycling rate [194], volume of WW [185], alkalinity [195,196], and turbidity and colour [188]. Models estimating target parameters (i.e. soft sensor readings) are then primarily developed for prediction of effluent properties as well as other parameters: BOD [197–199], COD [195,198,200], TSS [188,201], nitrogen [202,203] and phosphate [189,200,204] contents, turbidity [201,205], DO [206], E. Coli [188,207], as well as energy consumption [16] and a weather signal [208]. Among the examined soft sensors, several studies reported on-going developments while others are well-established full-scale industrial applications.

Mathematical models used for soft sensor development include multivariate linear methods such as PCA, PLS, MLR and other ML and AI methods including neural networks (NN), kernel machines, decision tree-based methods and autoregressive and time series models. Traditional statistical and multivariate linear methods are however considered not sufficient for such applications therefore adaptive nonlinear models and ANN, especially feedforward NN with backpropagation, should be preferred in soft sensor development to achieve higher accuracies [181]. Furthermore, the deep learning (DL) methods extensively reviewed by Alvi et al. [209] demonstrate superior capabilities not only in soft sensor development but also fault detections, including long short-term memory (LSTM) - a type of recurrent neural network (RNN) [210]. Aside from NN based methods, decision tree-based and adjunct statistical methods have high potential to improve soft sensor performance [94]. Recent popularity was gained by transformer models that are based on multi-head attention mechanism requiring less training than RNN [211,212]. The increased availability of algorithms and computational power, combined with continuous hardware input sensor accuracy and durability improvements are key in expanding soft

sensors in WW treatment sector.

In parallel, IoT technology is driving the development towards fully connected WWTPs. This interconnected network of sensors and actuators will enable RT data collection and analysis, resulting in more responsive and adaptive WW treatment processes. As extensively reviewed by Thakur and Devi [21], IoT sensor networks may include electrochemical sensors [213], electrical and optical sensors (absorbance, fluorescence, colorimetric, X-ray fluorescence (XRF), and chemiluminescence) and biosensors [214], as well as surface enhanced Raman spectroscopy (SERS), and SPR sensors and other devices. However, because of advancements in digitalization, traditional at-line methods such as titration, colorimetry, chemiluminescence, ion selective sensors (ISE), and voltammetry that are integrated into modern instruments also have a considerable potential for use in IoT networks. These methods can measure properties such as biological contamination (through portions of adenosine triphosphate (ATP)), toxicity, trace metals, as well as organics, inorganics, and nutrients [215].

Advanced data analytics and cloud computing will play a crucial role in managing big data from these sensors, facilitating RT decision-making and predictive analytics [216]. An important advance is the development of automated control systems that use IoT data to dynamically adapt WW treatment processes. This automation aims to optimize efficiency and reduce manual intervention, resulting in cost savings and lower error rates [217]. In addition, these technologies are expected to improve energy optimization and resource recovery in WWTPs and contribute to their transformation into water resource recovery facilities (WRRFs) [218]. However, these advancements also bring challenges in terms of data security [219], scalability and maintenance that need to be addressed for widespread adoption. Further integration of soft sensors and IoT technologies into WW management will significantly revamp the WWTPs RT monitoring and control [94]. In essence, the integration of soft sensors and IoT represents a step towards more efficient, accurate and sustainable WW treatment that aligns with broader urban sustainability and environmental protection goals [217].

6.2. Smartphone-based sensors

Smartphone sensors are emerging as cost-effective, user-friendly, and quick RT analyzers. Various studies showcase different sensors linked with smartphone apps. An integrated electrochemical device measured pH, free chlorine, temperature, pharmaceuticals, and HMs [220]. Metal ions like mercury, lead, chromium, nickel, copper, and iron were analyzed using a paper-based microfluidic device (μPAD), transforming the smartphone into a triple-indicator sensor [221]. Another μPAD, utilizing a smartphone camera, monitored hardness (calcium and magnesium ions), total phenols, and pH [222]. Additionally, water quality parameters such as nitrites, phosphates, and chromium were tracked via smartphone camera and analyzed with a reference model [223]. The device also processed colorimetric data for mercury concentration [224,225]. Furthermore, a miniaturized smartphone fluorescence microscope detected mercury, serving as both a signal collector

and image processor [226]. Recent literature, mostly within 2-3 years, indicates ongoing advancements in this field. Currently, the smartphone-based devices are not commercially available, and measurements are best performed on tap water or clear samples, as high solid concentrations hinder sensor performance [227].

6.3. Aquaphotomics

Aquaphotomics is an innovative field that utilizes mainly near-infrared (NIR) spectroscopy to analyze the interaction between water and light, revealing insights into water's structure and function in various systems [228]. This discipline examines water's absorbance patterns in the NIR region (traditionally in the area of first overtone of water i.e. 1300-1600 nm, but more recently also in other wavelength ranges) to study water content, hydrogen bonds, and hydration states in a range of applications [229], from WW treatment [230] and agriculture to pharmaceutical sciences [228]. Central to aquaphotomics is the "water mirror approach," which detects changes in water molecular networks due to physical or chemical perturbations, reflected in altered NIR spectra. The technique introduces water absorbance spectral patterns (WASPs), which capture these changes and serves as holistic markers in diverse applications. Key to successful aquaphotomics studies is the precise experimental setup, including high-quality spectral signals and specific instrument requirements, to accurately analyze and interpret the complex water species in aqueous systems [231]. While aquaphotomics presents a revolutionary approach, the industrial implementation of the technology is anticipated to be a gradual process due to the complexities involved in this advanced scientific field.

7. Conclusions

This review focuses on WW quality characterization and measurement technologies while including the main characteristics of WW, traditional laboratory-based assessments, an overview of RT process monitoring, and a future perspective on water quality control with emerging technologies.

The following conclusions can be drawn:

- WW quality characterization today is proven to be still ruled mainly by traditional laboratory-based measurements;
- The employment of sensors is getting more attention, and it is demonstrated that a vast number of parameters needed for a WW evaluation could be monitored through RT acquisitions;
- Advantages in the introduction of RT sensors throughout the treatment processes include faster, more frequent, and effortless ways of characterizing the WW, thus avoiding delays in time and sampling procedures;
- Currently, non-target screening measurements are the preferred solution to evaluate persistent compounds and hazardous chemicals;
- Breakthrough developments regarding modeling and optimization processes could be developed exclusively with the introduction of RT sensors within the WW treatment plant process control;
- Soft sensors employed to predict values of several WW target parameters as well as IoT technologies are gradually becoming established practices, with several examples reported, emphasizing their usefulness for a RT monitoring;
- Further advantages may derive from the implementation of new mathematical and statistical methods, such as AI and ML. Aquaphotomics methodology reveals its potential to extract important characteristics from NIR wastewater spectra;
- Future needs for WW quality characterization will be driven by the implementation of new legislative frameworks and new technological developments in analytical chemistry and sensors, that are becoming more accessible and affordable;

- Important roles will be also played by new scientific findings regarding pollutants and their potential effects on human health and the environment;
- Future works should be dedicated to the development of robust, long-lasting, and precise RT technologies allowing the detection of low concentrated pollutants with minimum sample preparation requirements. Economic and feasibility assessments should be considered before designing and implementing RT sensors *In situ*.

List of abbreviations

µPAD	paper-based microfluidic device
AES	atomic emission spectroscopy
AI	artificial intelligence
ANN	artificial neural network
ARBs	antibiotic resistant bacteria
ARGs	antibiotic resistance genes
BOD	biochemical oxygen demand
CECs	contaminants of emerging concern
COD	chemical oxygen demand
DAD	diode-array detection
DL	deep learning
DO	dissolved oxygen
DWW	domestic wastewater
E. Coli	escherichia coli
EM-W	electromagnetic wave
ET	electronic tongue
EU	European Union
FAAS	flame atomic absorption spectrophotometry
FRET	fluorescence resonance energy transfer
GC	gas chromatography
HMs	heavy metals
HPLC	high-performance liquid chromatography
ICP	inductively coupled plasma
IoT	internet of things
IR	infrared
ISE	Ion selective sensors
ISM	ion-selective membrane
LC	liquid chromatography
LOC	lab-on-chip
LSTM	long short-term memory
MFC	microbial fuel cell
ML	machine learning
MLR	multivariate linear regression
MPN	most probable number
MS	mass spectroscopy
N-DWW	non-domestic wastewater
NIR	near-infrared
NMR	nuclear magnetic resonance
NN	neural network
NNA	neural networks analysis
OES	optical emission spectrometry
ORP	oxidation-reduction potential
PCA	principal component analysis
PFAS	per- and polyfluoroalkyl substances
PLS	partial least squares
RM	resonant mirror
RNN	recurrent neural network
RPLC	reversed-phase liquid chromatography
RT	real-time
S&E	science and engineering
SDGs	sustainable development goals
SERS	surface enhanced Raman spectroscopy
S-ISM	solid state ion-selective membrane
SPR	surface plasmon resonance
TDS	total dissolved solids

TN	total nitrogen
TOC	total organic carbon
TP	total phosphates
TS	total solids
TSS	total suspended solids
UHPLC	ultra high performance liquid chromatography
UN	United Nations
USEPA	United States Environmental Protection Agency
UV-Vis	ultraviolet-visible
UWW	urban wastewater
VSS	volatile suspended solids
WASP	water absorbance spectral pattern
WRRF	water resource recovery facilities
WW	wastewater
WWTP	wastewater treatment plants
XRF	X-ray fluorescence

CRediT authorship contribution statement

Alessandro Moretti: Conceptualization, Methodology, Investigation, Data Curation, Visualization, Writing – Original Draft, Writing –Review & Editing. **Heidi Lynn Ivan**: Investigation, Visualization, Writing – Review & Editing. **Jan Skvaril**: Funding acquisition, Conceptualization, Methodology, Investigation, Data Curation, Vizualization, Supervision, Writing – Part of Original Draft, Review & Editing.

All authors contributed to the article and approved the submitted version.

Declaration of competing interest

The authors affirm that no commercial or financial relationships have influenced the research, thereby ensuring no potential conflicts of interest.

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This article does not involve data sharing as it did not generate any new data during the study.

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