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Review

The Application of Passive Sampling Devices in Wastewater Surveillance

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Abstract: Wastewater-based epidemiology (WBE) is a surveillance approach used to examine chemical and biological targets within a population. Historically, the most common approaches to wastewater sampling include grab sampling and composite sampling, which can be performed manually or using an automated sampler. However, there are inherent flaws with these sampling methods. They can miss analytes due to fluctuation events in wastewater and can have high cost and labour implications. Alternately, passive sampling is a technique that involves a sampling medium that can stay in an aqueous matrix for extended periods of time to provide a greater temporal coverage. This literature review examines the current passive sampling devices used in wastewater surveillance and the general contaminants they are targeting. The polar organic chemical integrated sampler, Chemcatcher®, diffusive gradients in thin films sampler and semipermeable membrane devices were among the most frequently deployed samplers in wastewater matrices. Chemical contaminants and pharmaceuticals were identified as the most common targets. Passive sampling of biological targets has received recent attention due to the surveillance of SARS-CoV-2; however, overall, there is a lack of critical knowledge relating to the deployment and associated variability of passive samplers used for biological targets. Notwithstanding, the ability of passive sampling to capture temporal fluctuation of analytes in wastewater make it a useful sampling technique for the surveillance of pathogens in the community. Future research should focus on addressing the gaps in knowledge to optimise the use of these sampling devices.

Keywords: passive samplers; wastewater surveillance; wastewater-based epidemiology

Citation: Shakallis, A.G.; Fallowfield, H.; Ross, K.E.; Whiley, H. The Application of Passive Sampling Devices in Wastewater Surveillance. *Water* **2022**, *14*, 3478. https://doi.org/10.3390/w14213478

Academic Editor: Christos S. Akratos

Received: 20 September 2022
Accepted: 28 October 2022
Published: 31 October 2022

1. Introduction

The ability to monitor a wide range of chemical and biological targets within a population is becoming an increasingly important global issue [1]. Wastewater sampling is a non-invasive technique that can be used to monitor environmental and public health impacts in the population through wastewater-based epidemiology (WBE) [2,3]. It can also be used to monitor the prevalence of pathogens in a population and identify potential hazards that may be excreted into the environment through the wastewater treatment process and reuse [4,5].

The premise of WBE is that extracts from the populations' urine, faeces and shedding events reflect consumption habits and infection/health within the population [6]. The first to postulate the idea that certain compounds found in wastewater can be linked to the increasing consumption of drugs in the population was Daughton [7]. Monitoring disease in the population is a component of public health; WBE is a complementary surveillance tool. As seen during the COVID-19 pandemic, WBE can be an effective early detection method of the presence of SARS-CoV-2 [8].

The accuracy of wastewater surveillance is impacted significantly by the sampling approach. Generally, samples are collected through grab or active sampling techniques, which can be performed either manually or using automated samplers. Grab sampling is a method that involves the collection of one-off spot samples from a sampling location at

pre-determined time periods [9]. Composite sampling, or active sampling, is a technique involving the collection of discrete samples taken at specified intervals [10]. A weakness of grab and active sampling accroaches is that they only capture information from the sampling site at the time the samples were taken [9]. Therefore, depending on sampling intervals and distance from the source of the target, spike events can still be missed in the sampling matrix [3,10,11].

Passive sampling is a diffusion based sampling technique that is increasingly used for monitoring a wide range of compounds in various matrices including water, soil and air [12]. Generally, the design of passive sampling devices includes a receiving phase in the form of a sorbent, filter and/or diffusion gradient with high affinity for the target analytes [13]. The process is based on the transport of the targeted analyte from the matrix being sampled, to the sampling devices' receiving phase [5]. These samplers can be left in the sampling matrix for an extended period (days, weeks or, in some cases, months), providing a greater temporal coverage, after which the time-weighted average concentration of analyte is determined [5,10,14–16]. Along with improved detection limits and pre-concentration of the target analyte for subsequent analysis, passive sampling can be less susceptible to contamination during transport, compared to handling of larger sample volumes [4,16–19].

Passive samplers used for microbiological collection in various water matrices are rare [20]. One of the earliest reported uses of passive sampling was the use of the Moore Swab developed by Moore [21]. The Moore swab, utilising medical gauze for microbiological sampling, was initially developed to monitor paratyphoid bacilli in a sewage outfall [21]. Gauze swabs were passed down through the drain covers into the flowing sewage and tied with sturdy twine to the drain covers [21]. The Moore swab was later adapted by Sattar and Westwood [22], for isolation of poliovirus types 1 and 3 in sewage; this technique was later implemented by de Melo Cassemiro et al. [23] for poliovirus isolation, this time in seawater.

These samplers are designed to follow Fick's first law of diffusion; though, due to conditions in wastewater matrices, Fick's first law of diffusion is not strictly followed [24]. There are two main configuration types in sampler design: equilibrium samplers and kinetic samplers. An equilibrium sampler will reach a quick equilibrium with contaminants in the aqueous media, which can happen either instantaneously or after some time, while a kinetic sampler will follow a time-integrated linear uptake. Depending on its properties, a target will be taken up by a sampler by means of either adsorption or absorption. Hydrophilic and hydrophobic contaminants/chemicals, pharmaceuticals, personal care products, antibiotics, licit and illicit drugs can all be targeted through the use of passive sampling devices [25].

Passive samplers have been used for many applications relating to WBE. For example, they are used as a forensic tool for law enforcement agencies to identify areas with high illicit drug activity [10]. The COVID-19 pandemic has led to an increased reliance of WBE for monitoring SARS-CoV-2 in populations. This has in turn led to increased interest in the use of passive samplers for biological targets [20,26–34].

While there is some information available regarding the use of passive sampling in water and wastewater microbiology, the depth of critical knowledge related to this area is limited. This literature review collates the current body of evidence relating to the use of passive samplers in wastewater matrices and the different types of samplers and target analytes. It also identifies the gaps in knowledge and areas for future research. The development of passive samplers is relevant for the monitoring and tracing of organics and emerging contaminants. The evaluation of passive samplers is also critical for the development of emerging monitoring strategies incorporated into future water management plans.

2. Materials and Methods

Articles included in this literature review were identified in August 2022 through Scopus®, Web of Science and Google Scholar using the search terms presented in Table 1. The snowball method was also applied to capture any additional articles that were missed during the initial search strategy. Articles were initially excluded if they were reviews

or not written in English. The articles were screened, and articles were then excluded if they did not sample wastewater or sewage. The reason for this exclusion was due to the possibility of ecological effects taking place by mixing effluent discharge and other aqueous matrices [35].

Table 1. All Keywords used to identify relevant literature on Scopus®, Web of Science® and Google Scholar®.

Search Terms Used to Identify Relevant Articles
"passive sample **"
AND
Wastewater OR sewage
AND
Exclude "reviews"

Note(s): ** Used when there may be possible variations of the search term.

3. Results

Following article screening, 96 papers were included for review. Sixty-three of the articles were field based, with only one article not including a field based component [36]. Twenty-two articles included both a laboratory and field component in their research article [9,12,18,24,25,37–53].

There were 40 passive sampler configurations found in the included articles (Table 2). Some of these configurations targeted chemical attributes—for example, polar [45,47,54–57] or non-polar [55,57,58]—others targeted chemical classes—for example, organic [17,45–47,54–56,59–63] or inorganic, [55]. In some articles, passive sampler configurations targeted more general analytes, for example, chemical compounds or toxins of concern found in wastewater [3,52,64–66].

Twenty-six articles targeted pharmaceuticals in wastewater treatment plants (WWTP) and sewage treatment plants (STP) influents and/or effluents [1,2,5,9,11,14–16,25,41,44,48,50,56,65,67–77].

Fourteen articles investigated passive samplers targeting endocrine disrupting compounds [9,19,37,43,44,49,50,60,78–84]. Six papers targeted illicit drugs [2,5,10,70,77,85]; however, of these papers only one focused on detection of one illicit drug, namely methamphetamine, in sewer pipes in an area with suspected illegal activity [10].

Seven articles targeted pesticides [9,16,18,46,56,65,86]. Micropollutants were targeted in four papers [59,60,87,88]. Per- and polyfluoroalkyl compounds were reported in five studies [40,42,51,89,90].

Other specific targets mentioned in only one or two papers in this review included: metals [55]; platinum group elements [12]; organophosphorus flame retardants [53,91]; optical brighteners [38]; nitrate and phosphate [24].

Twenty-five articles targeted biologicals including: antibiotic resistant genes [68]; biofilms [13]; paratyphoid bacilli [21,92]; *Salmonella* spp. [93–98]; *Mycobacterium tuberculosis* [96]; enteric organisms [99]; *Vibrio cholerae* [100]; and pathogenic viral genomes, including SARS-CoV-2 [20,22,27,28,30–34,36,68,96,101,102]. Alygizakis et al. [103] targeted biological contaminants but did not further define this term. In the context of this review, biological contaminants are understood as any biological organisms, including viruses and indicator microorganisms. Of the 25 articles, 11 articles were published prior to 1990 and 10 articles targeted SARS-CoV-2, leaving only four recent studies targeting biologicals using passive sampling techniques.

Table 2. Different types of passive samplers being used in wastewater surveillance and their targets.

Sampler	Sampling Device or Material	Configuration	Target	Reference
DGT [1,*]	Device	Binding agent: AG MP-1	Platinum group elements	[12]
		Binding agent: Chelex® and TiO_2	Metals	[55]
		Binding agent: Chelex® resin; filter: polysulfone membrane; diffusive agent: open pore gel	Silver nanoparticles	[104]
		Binding agent: mixed cation exchange gel; filter: nylon filter membrane; diffusive agent: polyacrylamide with agarose derivative cross linker gel	Melamine and related triazines	[105]
		Binding agent: mixed cation exchange gel; filter: PES, PTFE, PVDF, polycarbonate and nylon; diffusive agent: polyacrylamide gel	Denatonium benzoate	[106]
o-DGT [3]	Device	Binding phase: HLB binding gel Diffusive agent: agarose gel	Pharmaceuticals	[18]
		Binding phase: HLB binding gel Diffusive agent: agarose gel	Pesticides	[18]
		Binding phase: HLB containing binding gel Diffusive agent: diffusive gel	Polar organic contaminants	[54]
		Binding phase: HLB, XAD 18 or XDA-1 resin Diffusive agent: agarose gel Filter: PES membrane	Illicit drugs	[70]
		Binding phase: HLB, XAD 18 or XDA-1 resin Diffusive agent: agarose gel Filter: PES membrane	Antibiotics	[70]
		Binding phase: XAD18 resin Diffusive agent: agarose gel Filter: Hydrophilic Millipore membrane	Estrogen and estrogen-like compounds	[80]
		Binding phase: Sepra-ZT binding gel Diffusive agent: agarose gel	Pharmaceuticals	[16]
		Binding phase: Sepra-ZT binding gel Diffusive agent: agarose gel	Pesticides	[16]
		Binding phase: XAD 18 resin Diffusive agent: agarose gel Filter: PES membrane	Antibiotics	[14]
		Binding agent: XAD18 resin; filter: PES membrane; diffusive agent: agarose gel	Per and polyfluoroalkyls substances	[42]
		Binding agent: weak anion exchanger; filter: hydrophilic PES membrane; diffusive agent: agarose gel	Per and polyfluoroalkyls substances	[40]
		Binding agent: XAD18 resin; filter: membrane filter; diffusive agent: agarose gel	Estrogens	[43]
		Binding agent: porous carbon material gel	Antibiotics	[48]
		Binding agents: HLB, XAD18, or Strata-XL-A; diffusive agent: polyacrylamide and agarose gel	Endocrine disrupting chemicals	[39,79]
		Binding agents: HLB, XAD18, or Strata-XL-A; diffusive agent: polyacrylamide and agarose gel	Household and personal care products	[107]
		Binding agents: Sigma-MIP resin; filter membranes: PES, PTFE, PVDF, polycarbonate and nylon; diffusive agent: polyacrylamide	Fluoroquinolone	[108]
POCIS [2]	Device	Oasis HLB sorbent between two PES membranes	Pharmaceuticals	[1,9,11,25,56,65,67,69,71,72,75,76]
			Anticancer drugs	[74]
			Illicit drugs	[85]
			Fluoroquinolone antibiotics	[68]
			Antibiotic resistant genes	[68]
			Pathogenic viral genomes	[68]
			Polar organic contaminants	[54,62]
			Polar organic compounds	[55]
			Organic pollutants	[59,61]
			Hydrophilic contaminants of emerging concern	[57]
			Estrogens	[49,78,82,84]
			Methamphetamine	[10]
			Pesticides	[9,56,65]
			Endocrine disrupting compounds	[65]
			Chemical contaminants	[15]
			Beta-blockers and hormones	[9]
			Steroid hormones	[109]

Table 2. *Cont.*

Sampler	Sampling Device or Material	Configuration	Target	Reference
			Photosystem II inhibitors	[52]
			Emerging contaminants	[51]
		Immobilized ionic liquid between two PES membranes	Per-fluorinated substances	[52]
		Oasis HLB sorbent between two polysulfone membranes	Various emerging contaminants	[24]
		Triphasic admixture of hydroxylated polystyrene-divinylbenzene resin and adsorbent dispersed on a styrene divinylbenzene co-polymer	Pharmaceuticals and illicit drugs	[77]
		Home synthesized sorbents and nylon membranes	Organic contaminants	[63]
Chemcatcher®	Device	Teflon; diffusion limiting membrane; Ion-exchange receiving disk	Nitrate and phosphate	[24]
		SDB-RPS disks	Micropollutants	[88]
		HLB-L disks as receiving phase covered by PES membranes	Pharmaceuticals	[1,5]
		Receiving phase: SDB-XC, or SDB-RPS, or C18FF; diffusion phase: PES membrane or Omnipore membrane	Trace organic chemicals	[17]
		Empore disks	Estrogens	[82]
		C18 Empore disks	Organotin compounds	[3]
		SDB-RPS disk	Pharmaceuticals or pharmaceutical ingredients	[50]
		HLB disk covered by PES membrane	Personal care products	[5]
			Illicit drugs	[5]
		SDB-RPS disks	Estrogenic activity	[50]
SDB-RPS [4]	Material	SDB-RPS disks	Polar organic contaminants	[45]
		PES membranes	Estrogenic activity	[60]
			Organic micropollutants	[60]
		Empore disks	Estrogens	[82]
			Endocrine disrupting compounds	[19]
		SDK-RPS disks mounted on an aluminium alloy plate	Micropollutants	[87]
PASSIL [5]	Device	Ionic liquid between two PES membranes held between two screwed together plexiglass disks	Pharmaceuticals	[25]
MESCO [6]	Device	PDMS or PES or POM	Polar organic contaminants	[47]
Tampons	Material	Optical brightener free tampons	Optical brighteners	[38]
Hollow fibre silicone membranes	Material		Polar organic contaminants	[46]
Agarose hydrogel diffusion-based sampler	Material	HLB sorbent between diffusive hydrogel disks	Chemical and biological contaminants	[103]
		Strata-X SPE sorbent	Licit and illicit drugs	[2]
Microporous PE tube [7]	Material	Strata-X sorbent and agarose gel	Pharmaceuticals and personal care products	[2]
Multi-armed polyethene strip	Material		Industry discharge	[13]
SR sheets [8]	Material		Non-polar organic compounds	[55]
Polyethylene	Material		Poly- and per fluorinated alkyl substances	[89,90]
SPMD [9]	Device		Hydrophobic contaminants of emerging concern	[57]
			Chemical contaminants	[65]
			Pesticides	[65]
			Pharmaceuticals	[65]
		PE tubing	Pharmaceuticals	[69]
Silicone rubber	Material		Chemical characterisation	[66]
Silicone sampler	Material	Translucent silicone sheets	Hydrophobic organic compounds	[58]
MPS [10]	Device	Combined PDMS and Oasis HLB	Organic contaminants	[62]
Microporous ceramic sampler	Device	Diffusion phase: water membrane; reverse phase: Sepra ZT; retaining phase; pyrrolidone modified SDB polymer	Anticancer drugs	[73]

Table 2. *Cont.*

Sampler	Sampling Device or Material	Configuration	Target	Reference
Ceramic toximeters	Device		Dissolved dioxin-like PCBs	[37]
Polymer inclusion membrane	Material	NaCl receiving solution	Sulfamethoxazole	[41]
Partitioned based sampler	Device	Silicone rubber sheets	Chemical status (toxicity and chemical analysis)	[64]
HLB embedded cellulose acetate membrane	Material		Organophosphate flame retardants	[91]
Zetapore filter	Filter		Norovirus and ostreid herpesvirus type 1	[36]
Low-density PE	Material		Norovirus and ostreid herpesvirus type 1	[36]
Nylon nets	Material		Norovirus and ostreid herpesvirus type 1	[36]
Polyvinylidene difluoride immobilon	Material		Norovirus and ostreid herpesvirus type 1	[36]
Gauze pads	Material		Norovirus and ostreid herpesvirus type 1	[36]
Medical gauze	Material		Paratyphoid bacilli	[21]
			Polio virus	[22]
COSCa [11]	Device	3D printed acrylonitrile butadiene styrene hollowed sphere containing either electronegative filters, medical gauze, a cheesecloth or a cellulose sponge.	SARS-CoV-2	[28]
3D printed torpedo style sampler	Device	Contained medical gauze, swabs, electronegative filter membranes and cotton buds. Sampler wrapped in shade cloth.	SARS-CoV-2	[20]
3D printed matchbox style sampler	Device	Contained cotton buds, hot glued into location. Wrapped in shade cloth.	SARS-CoV-2	[20]
3D printed boat style sampler	Device	Contained medical gauze, swabs, electronegative filter membranes and cotton buds. Sampler wrapped in shade cloth.	SARS-CoV-2	[20]
Colander sampler	Device	Made from readily available colander from IKEA containing gauze swabs, electronegative filter membranes and cotton buds. Sampler wrapped in shade cloth.	SARS-CoV-2	[20]
Moore Swab	Material	Medical gauze	*Salmonella typhi* *Salmonella* spp. Paratyphoid B Enteric organisms *Vibrio cholerae* Coxsackievirus *Mycobacterium tuberculosis* SARS-CoV-2	[94–98] [93] [92] [99] [100] [96] [96] [27,102]
Organic cotton tampon	Material		SARS-CoV-2	[101]
3D torpedo style sampler	Device	Contained medical gauze, swabs, and electronegative filter membranes.	SARS-CoV-2	[101]
3D printed sampler	Device	Contained electronegative filters.	SARS-CoV-2	[30]
Cotton tampon-based sampler	Material		SARS-CoV-2	[31]
Ion exchange filter papers	Material		SARS-CoV-2	[31]
3D printed torpedo style sampler	Device	Contained electronegative membranes.	SARS-CoV-2	[32]
Zetapore membrane sampler	Material		SARS-CoV-2	[33]
Nylon membrane sampler	Material		SARS-CoV-2	[33]
3D printed torpedo style sampler	Device	Contained cotton swabs and electronegative filter membranes.	SARS-CoV-2	[34]

Note(s): * Binding agents, filters, and/or diffusive agents have been left out of this table when not specified in the referenced research paper [1] Diffusive gradient in thin films sampler [2] Polar organic chemical integrative samplers [3] Organic diffusive gradients in thin films sampler [4] Styrene-divinylbenzene reverse phase sulfonate [5] Passive sampling by ionic liquids [6] Membrane-enclosed sorptive coating [7] Polyethylene tube [8] Silicone rubber sheets [9] Semipermeable membrane devices [10] Mixed polymer samplers [11] COVID-19 sewer cage.

3.1. Sampling Sites

The majority of the articles in this review undertook laboratory studies to calibrate the samplers and/or determine sampling rates and diffusion coefficients. One laboratory study was included; while this work was not conducted at a wastewater or sewage plant, sewage water was spiked with a virus and uptake capacity was examined [36]. Twenty-five papers included a joint laboratory and field deployment study [9,11,12,18,24,25,31,37–53,108]. Where the main objective was not calibration of the sampler, the laboratory experiments included testing the optimal deployment time [18,37,38,40], the uptake capacity of different diffusion gradients [39,43,80], clogging [59], effect of pH, ionic strength and/or dissolved organic matter of the uptake of the target [43].

The majority of the studies (49) were conducted in WWTPs in Europe [1,3,5,11–15,24,25,31,33,36,38–41,44,47,49,50,52,55,56,58–62,64–67,72,73,77,78,80–84,87,88,92,95,103,107,109]. Twenty-one studies deployed passive samplers in WWTPs in North America [4,9,10,16,18,27,28,30,34,54,57,69,71,85,89,90,93,94,96,98,100], while twelve articles reported testing in Asia [42,43,48,51,53,63,70,80,91,102,105,106,108]. Six studies were conducted in Australia [2,17,19,20,32,101]; of these six studies, one sampled in Antarctica and transported samples to Australia where analysis took place [17]. Four papers sampled in South America [68,74,76,97]. Two papers sampled in South Africa [37,75] and one sampled in Egypt [46].

Seasonality of the deployment was not specifically considered in any of the reviewed articles; however, one study did include a component where the temperature of the sample matrix was recorded to consider the influence of temperature on the uptake of target analytes [51]. Hoque et al. [69] sampled in both summer and autumn; while the highest accumulation of pharmaceuticals was observed in autumn, the authors concluded that this may not be due to the temperature difference but the WWTP treatment and removal. Other articles mentioned the effects of temperature; however, Wang et al. [51] was the only study to have extensively conducted batch experiments to analyse temperature impact on uptake capacity of per fluorinated substances by the POCIS. Other studies analysed deployment time, and in both instances, it was concluded that an increase in deployment time and/or temperature, increased the chemical target uptake by the sampler [36,37,44,46,51].

3.2. Passive Sampling Devices Used in Wastewater Surveillance

Briefly, the most common passive samplers that have been reported are the polar organic chemical integrative sampler (POCIS), the Chemcatcher® and diffusion gradients in thin films (DGT) samplers (Table 2).

3.2.1. The Polar Organic Chemical Integrative Sampler (POCIS)

The POCIS is among the most studied passive sampling device, with 38 articles using the device (Table 2). Generally, these devices are used for targeting hydrophilic molecules, including pharmaceuticals, personal care products, and endocrine disrupting compounds [4,15,44,84]. The general configuration of a POCIS is a Hydrophilic-Lipophilic Balance (HLB) sorbent between two polyethersulfone (PES) hydrophilic membranes and stainless-steel mounts to hold membranes in place (Figure 1). Essentially, chemicals can adsorb to the HLB sorbent phase after diffusing through the PES membranes, and following deployment are extracted from the sorbent phase [81]. The PES membrane used in POCIS has a low tendency for biofilm development [4].

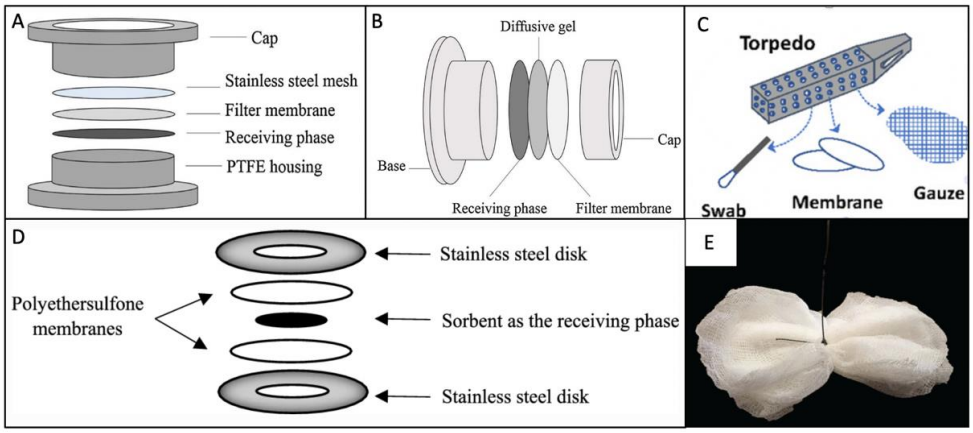

Figure 1. Passive sampling devices that have been utilised in wastewater surveillance. (**A**) Representation of Chemcatcher passive sampler configuration comprising of outer housing units containing a stainless steel mesh, filter membrane and receiving phase [110]; (**B**) Representation of a Diffusive Gradients in Thin Films (DGT) passive sampler comprising of outer housing units holding together a filter membrane, diffusive gel and receiving phase [110]; (**C**) Configuration of a 3D torpedo-style passive sampling device designed for sampling of SARS-CoV-2 containing swabs, medical gauze and membrane filters [101]; (**D**) Representation of a Polar Organic Chemical Integrative Sampler (POCIS) configuration comprising of a receiving phase sorbent between two polyethersulfone (PES) membranes held together by two stainless steel disks [111]; (**E**) The Moore Swab comprised of medical gauze pads held together with string [112].

POCIS samplers, like other passive samplers, can be designed for a particular target; however, POCIS samplers are configured quite differently depending on target analytes, pharmaceuticals, pesticides, or chemicals [15,44]. The $POCIS_{chem}$, $POCIS_{pesticide}$ and $POCIS_{pharm}$ configurations differ in the sorbents sandwiched between the two PES membranes. For example, the $POCIS_{pharm}$ will typically contain the general HLB sorbent [5,10,44], while the $POCIS_{pesticide}$ will generally contain a mixture of three sorbents [44]. While the $POCIS_{pesticide}$ has been reported to have better uptake rates than that of $POCIS_{pharm}$, there are benefits to using the $POCIS_{pharm}$ configuration. For example, the pharmaceutical configuration that it is less suspectable to rupturing than the pesticide configuration. The $POCIS_{pesticide}$ configuration tends to take up more water risking the membrane as there is an increased rupture susceptibility [44].

3.2.2. Diffusive Gradients in Thin Films (DGT) and Organic-Diffusive Gradients in Thin Films (o-DGT)

Nineteen articles used DGT and o-DGT samplers to monitor their target analytes (Table 2). Diffusive Gradients in Thin films (DGT) and organic-Diffusive Gradients in Thin films (o-DGT) are typically used to monitor a range of targets, including but not limited to nutrients, metals, and organic and inorganic compounds in various matrices [12,40]. The typical configuration of DGT samplers is a binding layer, diffusive gel, and filter membrane (Figure 1). The binding layer is responsible for the uptake of the analytes being targeted, the diffusive gel facilitates the diffusion of the targets, and the filter membrane is added for the protection of the gel layers. A standard plastic moulding surrounds and houses the sampler [40].

This is a more recent passive sampler that was developed on the basis of Fick's first law of diffusion and can avoid potential sample transportation and grab sample pre-treatment errors [40]. In comparison with other passive samplers, o-DGTs most often reportedly reflect concentrations similar to grab samples taken over the sampling period [16]. o-DGT devices can also accommodate variable temperatures in the sampling matrix [16]. Different resins are often tested with DGT samplers. HLB, also used for POCIS, has been

is recommended for DGT in wastewater sampling due to its robustness in environmental conditions [107].

3.2.3. Chemcatcher®

Eleven articles used a Chemcatcher® device (Table 2). Chemcatcher® passive sampling devices generally contain styrene-divinylbenzene absorbent bound in a polytetrafluoroethylene (PTFE) matrix disk (Figure 1) [5]. Like the POCIS, HLB disks are also used in Chemcatcher® as the binding agent [5]. The use of disks in Chemcatcher® devices minimise risk as the sorbent is immobilized and field data variability is reduced [5].

3.2.4. Semipermeable Membrane Device (SPMD)

In the early stages of passive sampling device research, SPMDs were developed for the purpose of targeting non-polar contaminants [44,84]. The general design of SPMD is a tubular polyethylene membrane containing triolein [52,69]. These devices showed relationships with both grab and auto-sampling and became a possible alternative to traditional sampling methods [84]. Hydrophobic organic chemicals can be taken up quite well with an SPMDs and therefore these devices are often preferred when the analyte being targeted is hydrophobic [44,57]. SPMDs are often spiked with performance reference compounds to aid in calculating the target sampling rate [65,69].

3.2.5. Other Sampling Devices

Ceramic based passive sampling devices consisting of a porous structure allowing for diffusion of the chemicals are used for polychlorinated dibenzo-p-dioxins, polychlorinated dibenzofurans (PCDD/Fs), dioxin-like polychlorinated biphenyls, poly aromatic hydrocarbons, and volatile organic contaminants [37]. Various membranes have been used for virus uptake; these include Zetapor® filters, low-density polyethylene, nylon nets, polyvinylidene and electronegative filters (Table 2). The Moore swab was the most common sampler for biologicals in the mid to late 1900s (Figure 1). More recently electronegative membrane filters are most commonly used in virus uptake, specifically for SARS-CoV-2 sampling (Table 2) [20,28,30,34,101].

3.3. Method Design

Optimal deployment time was assessed in twelve articles [14,15,18,39,40,50,51,59,73,80,83,89]. These articles all noted that it is difficult to establish a universal optimal deployment, due to all analytes being tested having different molecular structures and respond differently to competing compounds. It is also important to note that each sampler is configured and designed differently; so, while these papers identify a difficulty in establishing a universal optimal deployment, this will change depending on the sampler being used and the analyte being targeted. For example, some compounds may degrade or break down on the filters after 4 h and others after 192 h due to dissolved organic matter in the sampling matrix [73]. Dissolved organic matter can affect sampler uptake resulting in inconsistencies between laboratory and field results, specifically mentioned when sampling using DGT devices [12,18,39,43]. Chen et al. [107] reported dissolved organic matter having no effect on DGT measurements, but recognised that dissolved organic matter made it difficult for compounds bound to the samplers to pass through the diffusive layer due to binding of compounds to dissolved organic matter in the wastewater.

As well as deployment time, batch experiments have also been reported by Wang et al. [51] to analyse the effect of temperature on the sampling rates of perfluorinated substances by the immobilised ionic liquid (IIL) device. The sampling rate was tested at 10, 25, and 35 °C; there was a slight increase in sampling rate as the temperature increased [51]. Many laboratory experiments involved calibration of the sampling devices before field deployment, without focusing on any one possible limitation or effect. However, due to differences in laboratory and field conditions, many times results were quite different from the predicted values.

3.4. Comparison to Grab Sampling

Thirty-six studies included in this review compared the results from passive samplers to those acquired through grab or composite sampling. The findings from these studies showed that the recovery from grab, composite and passive sampling was dependant on the target. Cristovao et al. [68] and Vallejo et al. [83] both reported that grab samples, collected at various stages throughout the year missed a high number of occurrence of antibiotics when compared to the results obtained using the POCIS. The same was not found by Tan et al. [19] and Zhang et al. [52] who both found grab samples had a higher recovery in endocrine disrupting compound detection than passive samplers. Petrie et al. [5] reported that the Chemcatcher® detected the micropollutants benzophenone-1, dihydromorphine and ketamine, which were missed when wastewater was collected by composite samplers. It was argued that the increased sensitivity supports the use of passive samplers for quantitative analysis [10]. Alygizakis et al. [103] also reported 35 of the compounds detected by passive sampling were missed by composite samples.

From the articles reporting a comparison between passive and grab sampling techniques, many found good agreement between the two sampling methods when sampling for endocrine disrupting chemicals [14,39,80,83], organic contaminants [52,54], pharmaceuticals [4,15,67,68] and viral genomes [68]. Rafiee [102], compared grab, composite and passive sampling for SARS-CoV-2; this study reported good agreement between the Moore's sampler and composite samples, whilst the grab samples under reported the presence of the virus. Due to the requirement of power sources for active sampling, labour costs and complexity, passive sampling methods are preferred as a feasible alternative, where appropriate [14,19,42].

3.5. Extraction Techniques

Extraction of the targets from the sampling devices varied depending on the sampler and target analyte. Eight articles used extraction methods involving the sorbents being eluted through solid phase extraction [37,51,55,62,69,81,82]. Seven articles included a rinse cycle using an extraction solvent [5,19,45,52,61,83]. Eleven articles extracted the recovered samples through elution and evaporation techniques [4,5,16,19,24,45,52,53,61,68,83]. Rujiralai et al. [49] and Vermeirssen [109] reported extraction through glass wool, followed by an elution step. Cristovao et al. [68] and Vincent-Hubert et al. [36] both used nucleic acid extraction kits as they targeted viral fragments from norovirus and ostreid herpesvirus type 1. Hayes et al. [28] and Schang et al. [20] both used RNA extraction of samples captured on the filters and other sampling materials placed inside the passive sampling devices deployed in wastewater, followed by rt-qPCR for SARS-CoV-2.

Other extraction techniques included the use of ultrasonic baths [3,14,73] and automated extraction units [80]. The automated extraction unit used in Guo et al. [80] decreases the amount of solvent reduced during the extraction process. Vrana et al. [58] reported the use of an extraction gel column and evaporation with a gas flow. McKay et al. [2] extracted the targeted pharmaceuticals in polypropylene tubes, followed by sonication and evaporation. Polypropylene tubes were also used by Trommetter et al. [12] to extract platinum group elements, followed by elution in a HCl/thiourea mixture.

3.6. Analysis of Extracts

Analysis of the sampler extracts varied and was dependant on the target analyte. Generally, the most common analysis technique for chemical targets, after passive sampler extraction, was liquid chromatography coupled with mass spectrometry (LC-MS), with 38 research papers utilising this method [1,2,4,5,9,10,14–18,24,25,39,40,42,45,48,50,51,53,54,57,59,62,64–67,69–73,81,82,87,103]. Gas chromatography coupled with mass spectrometry (GC-MS) was used in nine articles [3,19,47,49,52,57,58,61,83,89]. Other analytical techniques reported include inductively coupled plasma mass spectrometry (ICP-MS) [12]; high performance chromatography with diode array detector (HPLC-DAD) [41]; inductively coupled plasma optical emission spectrometry (ICP-OES) [13]. One article sampling for optical

brighteners used UV light to determine the presence of the brighteners on the passive sampler [38].

Ten articles analysed sampler extracts through bioassays; commonly CALUX (Chemical Activated LUciferase gene eXpression) bioassays or the yeast estrogen screen assay [17,37,43,55,56,68,80,84,88,109]. Other techniques of interest included: DNA/RNA extraction followed by qPCR for analysis of biological contaminants [103]; reverse transcriptase-qPCR (RT-qPCR) for analysis of viral RNA fragments [20,28,36]; and qPCR for viral DNA detection [36].

4. Discussion

4.1. Use of Passive Samplers in Wastewater

Passive sampling is an effective technique used in wastewater surveillance both for environmental protection and WBE purposes in that there is a large range of target possibilities, the ability of target specific deployment and no need for a power source. Passive sampling can provide time weighted average concentrations, can catch temporal fluctuations of the target analyte and the samplers can be designed for specific targets, increasing sensitivity [14]. The time weighted average concentration calculated with passive sampling means the measurements obtained are more representative of the sampling period [15]. Though the time weighted average can also be obtained with composite sampling, the removal of a power source necessity with passive sampling makes it an appealing alternative. While the literature has reported similar concentrations are obtained between passive sampling and grab sampling, the main advantage of passive sampling is the simplicity, ease, low cost and no requirement for a power source [19].

There is a vital need to develop passive samplers aimed at targeting emerging contaminants of concern and to ensure significant contaminants impacting the environment and human health are not overlooked [55]. Environmental protection is a driving factor in wastewater sampling [13,51]. Accurate assessment for risk management is required for managing water quality [113]. Furthermore, there is a need for monitoring of certain contaminants or substances to determine their fate once released from treatment plants if not treated effectively [16,68]. While the concentrations of most chemicals are reduced through a treatment plant, there are many organic pollutants, micropollutants and transformation products still released into receiving ecosystems [60,103].

This literature review identified that there is a lack of research evaluating the use of passive samplers for monitoring and tracking biologicals. Prior to the COVID-19 pandemic, only four articles targeted biologicals in the 2000s [13,36,68,103]. Since the pandemic, there has been a rise in the number of reports utilising passive sampling for detection of SARS-CoV-2 in the wastewater. Due to the gap in the literature, samplers designed for viral uptake have not been optimised. This has implications for SARS-CoV-2 monitoring [70,114]. Schang et al. [20] and Hayes et al. [28] have both developed a passive sampling unit, not only for detection and surveillance of SARS-CoV-2, but also for a broader use in wastewater-based epidemiology. Due to temporal fluctuations and shedding events in the community, passive sampling can potentially ensure these events are captured and detected, making it an attractive sampling method for biologicals [3,10,115]. Further study into better materials for SARS-CoV-2 adsorption has recently been reported [29,31,33,101].

4.2. Current Limitations in Passive Sampling

Passive sampling is not without its limitations. These limitations include both environmental factors and passive sampler design. The applicability of passive sampling is still not demonstrated beyond doubt as it still faces many challenges [116]. Furthermore, there is a difficulty in overcoming the impact environmental conditions have on target uptake [19].

Fouling of samplers during deployment is a challenge of passive sampling and its effect on uptake is not yet completely understood [117]. Due to samplers being left in various aqueous environments over an extended period of time, samplers are exposed to microbial presence leading to biofilm formation and fouling [118]. Fouling on the samplers is a problem that is not faced with grab sampling. Fouling of passive samplers may interfere

with the sampling of the target analyte; however, the extent of the potential bias introduced is still unknown [117–119]. Current understanding suggests that the growth of biofilms and fouling of samplers can interfere with recovery by impacting diffusion due to pore blockage and cause a resistance to mass transfer [120]. A greater understanding of the influence of membrane and sampler fouling on analyte uptake is needed [11].

Competitive binding is a major limitation in passive sampling. It can be caused by dissolved organic matter or other chemicals, or contaminants present in the sampling matrix [53]. Competitive binding is one explanation for when a plateau or decline is observed in the accumulated analyte. Another explanation is a breakdown of the target analyte during the deployment [39,73]. Alternatively, the sampler may have simply reached equilibrium. The main driver reported in the literature for competitive binding is dissolved organic matter [12,39]. Dissolved organic matter can affect the equilibrium and kinetics of adsorption for compounds through either blockage or site competition. This is shown in a decrease of uptake after a peak [39].

While a time weighted average can be provided, the fluctuating concentrations of analytes can cause variable uptake rates due to environmental factors, such as temperature and pH, and desorption [19,45]. The reliability of passive sampling can be influenced by the setup and calibration of the sampling device, chemical analysis, varying environmental conditions, such as flow rate, temperature and pH, and fluctuating concentrations [12,40,45,69,86]. Designing a passive sampler for a specific target analyte improves the sensitivity of the sampling technique.

Time series experiments were included in a few papers measuring the uptake of chemical and pharmaceutical analytes by samplers, mainly including the POCIS and DGT. There is a lack of literature investigating uptake rates of biologicals by the samplers in wastewater. To our knowledge, Hayes et al. [30] is the only research article to have investigated viral uptake, namely SARS-CoV-2, over a period of time. While their time series had a maximum of 50 h, there is no understanding of uptake for a longer period and these results cannot be compared with another research paper. This highlights another gap in the literature, not only there being a limit to passive sampling of biologicals, but also there being a lack of literature understanding the update of biologicals by the samplers and how this can be impacted by environmental conditions.

Many articles report a combination of laboratory and field deployments. The uptake experiments that are conducted in a laboratory setting in a flow through system are highly controlled [17]. The environmental factors and concentration peaks that occur in a WWTP are not easily replicated in a laboratory environment, and this can account for observed differences between laboratory and field studies [17].

Another limitation identified with the design of passive samplers is the thickness of the diffusive boundary layer which can impact the mass transfer of chemicals [14]. The thickness of the diffusive boundary layer has been investigated and poor choice of the boundary layer has the potential to lead to measurement discrepancies [18,89].

4.3. Sampling Method Design

Most articles comparing grab and passive sampling, report the concentrations recovered are in agreement [4,14,15,52,54,68,83]. Notwithstanding, continued use of both grab and passive samples would ensure a well-rounded sampling technique for monitoring while passive sampling is still being developed. While composite sampling can provide a time weighted average, much like passive sampling, this method can be costly and dilute the target causing detection failures, especially when dealing with low concentrations [29]. Composite sampling shows similar values to passive samplers; however, the increased cost and detection failures make passive samplers more favourable [11]. Due to composite sampling essentially being a mixture of grab samples, spike events can still be missed if the sampling intervals are not selected correctly [11].

There is an increasing number of passive sampling devices being used in aqueous matrices, each with different specialisations, as described above [13]. Mechelke et al. [121], identified the parameters for an ideal passive sampling device for uptake in an aqueous

matrix. An ideal device would not be affected by hydrodynamic changes, would accumulate a sufficient concentration of the analyte required for laboratory analysis and respond to environmental concentration fluctuations during deployment. While a device of this sort has not yet been developed, the main goal of a passive sampler is to select the best sorbent, membrane and/or resin that will take up the target compound [17].

The purpose of calibration experiments in the laboratory prior to field deployment is to determine the sampling rate (R_s) or the diffusion coefficients [15,39]. While the R_s determined in laboratory experiments is rarely met in field studies, due to the inclusion of unprecedented environmental conditions, the sampler's general efficacy can still be predicted [3,13,16,54].

During the calibration, testing different temperatures and or pH conditions also gives another good indication of the abilities of the samplers being used [44,69]. Again, due to fluctuating and unpredictable environmental conditions, results of calibration experiments do not always match the field experiments.

4.4. Sampling Extraction and Analysis

The accuracy and efficacy of passive samplers is influenced by the recovery methods used. The methods of dislodging microbes or desorbing chemicals may alter in robustness, in turn affecting recovery [122]. When it comes to biologicals, recovery includes a series of physical shaking actions to dislodge the sample followed by subsequent analysis. The efficacy of recovery methods can be analysed through qPCR/RT-qPCR or culturing. Culturing will determine the concentration of viable pathogens sampled, while PCR will detect any DNA/RNA fragments of the pathogen. PCR can overestimate the number of cells in a sample and does not distinguish between dead and viable cells. Furthermore, the efficacy of the DNA/RNA extraction is dependent on both the sample matrix and the methodology followed. Likewise, the extraction and analysis techniques used for chemicals and pharmaceuticals can differ in sensitivity.

POCIS sampler extraction methods were found to be quite similar across the literature. The results obtained in these studies were varied due to the types of chemicals and pharmaceuticals targeted; however, when comparing results from each study with another targeting the same analyte, there was agreement between the findings. The high number of papers found in the literature targeting chemical entities in wastewater means trends between papers can be identified. The similar extraction and analysis techniques benefit this.

4.5. Future Studies

Further studies could involve creating more robust samplers that are less influenced by their environment when deployed in the field. There are already reports of the o-DGT passive sampler being less influenced by the surrounding environment, having the ability to account for temperatures and flow conditions in the field with adjustable sampling rates [16,54].

The differences in uptake between calibration and field deployment still need to be further explored; however, a focus on uptake trends and variations in sampling rates can generate the information required to develop a stronger understanding of the use passive sampling for WBE.

Development of optimal passive samplers for biologicals and other emerging contaminants are needed in future studies. While there is a move towards passive sampler use in SARS-CoV-2 detection, investigations considering the optimal uptake of this, and other viruses should be considered, as well as further understanding of uptake over the deployment time. It is likely that there will be an increase in passive samplers used for pathogen surveillance in a population.

5. Conclusions

This literature review identified 96 articles utilising passive sampling as a technique to monitor the presence of various target analytes in wastewater or sewage. The most commonly used sampler configurations included the POCIS, DGT, Chemcatcher® and SPMD samplers. Chemical contaminants and pharmaceuticals are currently the highest targeted analytes in wastewater. This identified a gap in the research regarding the use of passive samplers for surveillance of biological material, particularly bacteria and viruses. This is particularly relevant considering the increasing significance of WBE for the monitoring of SARS-CoV-2 during the COVID-19 pandemic. There is still much improvement to be made on the method design for passive samplers, including the configuration of a more robust sampling device, less impacted by temperature, pH, and flow rate of the deployment environment. Overall, passive samplers are as effective as grab sampling, with the added benefit of providing a time weighted average, higher sensitivity to analytes and pre-concentrated samples. Other added advantages of passive sampling as opposed to active or grab sampling are the power and overall labour costs. With further investigation in optimal passive uptake techniques, passive samplers provide an attractive sampling alternative in wastewater matrices.

Author Contributions: A.G.S., H.W., H.F. and K.E.R. designed and participated in review design. A.G.S. drafted and edited the manuscript. H.W., H.F. and K.E.R. corrected and contributed to the manuscript. All authors have read and agreed to the published version of the manuscript.

Funding: This research received no external funding.

Data Availability Statement: Not applicable.

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Conflicts of Interest: The authors declare no conflict of interest.

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