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Brackish groundwater desalination by reverse osmosis in southeastern Spain. Presence of emerging contaminants and potential impacts on soil-aquifer media

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ABSTRACT

Desalinated brackish groundwater is becoming a new source of water supply to comply with growing water demands, especially in (semi-) arid countries. Recent publications show that some chemical compounds may persist in an unaltered form after the desalination processes and that there is an associated risk of mixing waters with different salinity for irrigation. At the university of Alicante campus (Spain), a mix of desalinated brackish groundwater and water from the existing aquifer is currently applied for landscape irrigation. The presence of 209 emerging compounds, surfactants, priority substances according to the 2008/105/EC Directive, 11 heavy metals and microbiological organisms in blended water and aquifer samples was investigated. Thirty-five compounds were detected (pesticides, pharmaceuticals and surfactants) among them two priority substances α-endosulfan and Ni were found above the permitted maximum concentration. Blended water used for landscape irrigation during the summer period is supersaturated with respect to carbonates, which may ultimately lead to mineral precipitation in the soil-aquifer media and changes in hydraulic parameters.

Keywords: Brackish groundwater; Emerging contaminants; Reverse osmosis; Aquifer-soil impacts; Water quality

1. Introduction

The availability, quality and quantity of water resources are going to be of the most important problems to face in the decades to come. A shortage of water imposes constraints on economic, social and human development, and may even cause severe damage to ecosystems if water abstraction rates exceed natural renewal rates [1]. To meet the growing water demand, new sources of water are required, and the desalination of both seawater and water from brackish aquifers with chloride concentrations between 300 and 10,000 mg L^{-1} [2] is one of the most extensively used ways worldwide to reach this goal [2,3]. There are examples of desalinated water supplementing water supply, e.g. in the Spanish Islands [4],

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Israel [5] and the Far East [6], for irrigation [7] or industrial processes.

Reverse osmosis (RO) desalination plants in coastal areas are common in many parts of the world. Desalination technologies applied to brackish aquifers constitutes an important strategy to increase water supply in coastal and inland areas, providing a new and previously disregarded resource. Among the advantages to be considered, brackish water contains less salt than marine water, implying a lower energy demand, being economically more cost-effective due to a reduced treatment cost [3]; however, apart from reject disposal, some environmental issues also need to be addressed.

RO desalination processes remove salts, but some other chemical compounds may persist in an unaltered form. Among them, a group called emerging pollutants, a number of regulated and nonregulated pollutants such as pharmaceuticals (Phs), personal care products, illicit and licit drugs, and surface active substances (surfactants) have introduced a new challenge to water quality problems [8]. Many of them are known to behave as endocrine disruptors [8–12], an exogenous agent which interferes with the synthesis, secretion, transport, binding action or elimination of natural hormones in the body that are responsible for the maintenance of homeostasis, reproduction, development and behaviour. Over the last few years, an increasing number of investigations have raised the concern of the presence of these chemicals in the natural environment [8–11], since their effects on biota and human beings may be either dangerous or unknown even at low concentrations ($\text{ng}\,\text{L}^{-1}$ to $\mu\text{g}\,\text{L}^{-1}$).

All emerging contaminants have the common feature of not needing to persist in the environment to cause negative effects, since their high transformation rates or removal by wastewater treatment plants can be nullified by their continuous introduction into environment. Their complete removal by conventional wastewater treatment plants is not yet clear [8–10], and their presence in water has mainly been detected in sewage treatment facilities [8,9,11–13]. Although, they are not routinely monitored due to a lack of regulations in force and unavailable analytical methods, their presence in water is undesirable, even if compounds have low or acute toxicity.

A second constraint to be considered refers to the suitability for irrigating with a blend of desalinated and surface or groundwater due to possible associated impacts on plants, soil and aquifer media [14,15]. Mixed waters with different salinities can lead to the formation of unexpected chemical precipitates [16]. The use of desalted groundwater for irrigation has potential drawbacks [17], such us changes of hydraulic properties of soil-aquifer systems, e.g. hydraulic conductivity or porosity, as a consequence of mineral precipitation [18,19]; root growth blockage and plant uptake of pollutants [20,21]; as well as leaching of contaminants to groundwater [22,23].

At the university of Alicante (Alicante, Spain), a blend of desalted and raw groundwater from a brackish aquifer is currently used for campus landscape irrigation [24]. The desalination treatment consists in a RO process carried out at a small scale plant located onsite.

The aim of this study was: (i) to investigate the occurrence of 209 emerging pollutants, priority substances as defined in the 2008/105/EC directive [25] and heavy metals, in the aquifer and in the mixed desalted water used for irrigation, (ii) to assess the quality of water being used for landscape irrigation, and possible future impacts on soil-aquifer media due to its application.

2. Study area

The study site (Fig. 1), located in southeastern Spain, is characterised by a semi-arid Mediterranean climate (18°C average annual temperature) with a low annual precipitation ($300\,\text{mm}\,\text{yr}^{-1}$) distributed in several uneven events. Surface perennial streams rarely exist and watercourses only operate during heavy rainfall events producing important flood episodes. The region counts with an important water demand, being groundwater the most significant component of the water resources.

A quaternary unconfined aquifer of detritic origin with an average thickness of 16 m and a total extension of $81\,\text{km}^2$ outcrops in the study area [26]. This aquifer is directly recharged by precipitation and irrigation returns under cultivated zones. Due to its low exploitation, groundwater levels do not show important changes, ranging between 11 and 19 m below ground surface. The regional flow direction is from northwest to southeast, where the aquifer discharges to the Mediterranean Sea. A few seasonal springs and seepage areas are also discharge points of the aquifer. The aquifer is mainly composed by silts and sands, with a very low presence of clays, overlying the impervious loam of Cretaceous age. Imbedded thin gypsum layers are frequently found in outcrops and in drilling logs. As a consequence of the geological background, groundwater presents a high natural salinity, making it unsuitable to be used as drinking water or any other purpose, and therefore requires desalination treatment for use. Groundwater electrical conductivity (EC) values are $\sim 7{,}500\,\mu\text{S}\,\text{cm}^{-1}$, hardness

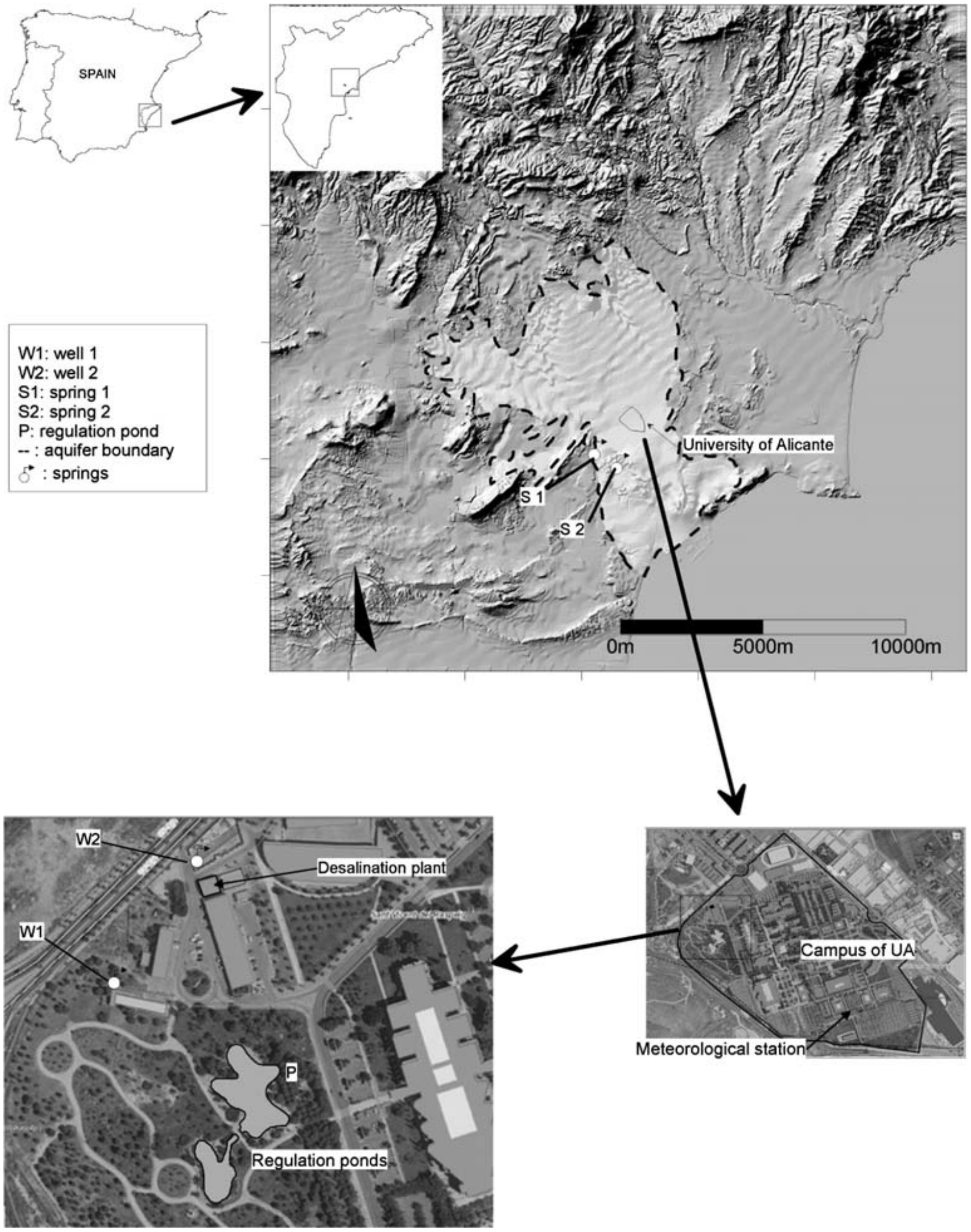

Fig. 1. Study area and aquifer boundary. Location of the defined sampling points.

above 1,900 mg L^{-1} as $CaCO_3$ and high concentrations of chloride, 1,500 mg L^{-1}; sulphate, 1,700 mg L^{-1} and sodium 1,300 mg L^{-1}.

The study was performed at the university of Alicante campus, which extends over an area of approximately 100 ha of vegetated land above the aquifer. At the study site, the groundwater level ranged between 11 and 13 m below ground surface for the studied period. There is a significant land cover extension mainly made up of Mediterranean plants such as *Pinus pinea, Platanus hispanica, Juniperus phoenicea*, etc., and due to the region structural water scarcity, irrigation is carried out with a mixture of different fractions of desalinated brackish groundwater and water from the aquifer. Vegetation is drip and sprinkler irrigated with water from a storage pond integrated in the campus landscape design. Water from the aquifer is pumped directly from 2 wells located in the campus that reach 33 m depth, screened over the entire aquifer thickness (Fig. 1). Groundwater pumped from well W1 is directly stored in the pond; water abstracted from well W2 is desalted in the RO desalination plant and conveyed to the pond, where it is blended with raw groundwater for irrigation purposes. The proportion of raw groundwater mixed with the desalted water is variable and ranges between 5% for winter and 22% during summer. Rejected brines are conveyed through a pipeline to an ephemeral creek in the north-western part of the campus.

2.1. The brackish water desalination plant

In 1997, the RO desalination plant located in the campus (Fig. 2) came into functioning in order to treat brackish aquifer water for the purpose of landscape irrigation. The plant was initially set up for academic purposes. Its treatment capacity is currently of 450 $m^3 day^{-1}$. The conversion capacity, which is the portion of the volume of desalinated water respect the volume of feed water, of the plant is 72% due to the high concentration of sulphates present in the brackish groundwater.

Treatment consists of a single line of 25 membranes distributed into two stages, with 15 in the first stage and 10 in the second one. The 8040-UHY-ESPA membranes, *Hydranautics*®, applied are spiral-wound, aromatic polyamides arranged in modules. A working pressure of 1,200 kPa is controlled by a variable frequency device. For a further description of the desalination plant, the reader is referred to [24].

Pretreatment consists of both sand and cartridge filtration with a continuous application of 3.8 $mg\,L^{-1}$ PERMATREAT191 antiscalant manufactured by Houseman, UK [27]. PERMATREAT191 is a sodium salt of the aminotrimethylenephosphonique acid N $(CH_2PO_3HNa)_3$, the active compound accounts for 50% [28]. No additional chemical pre- and posttreatment is carried out due to the quality of the feed water and its final use.

After groundwater desalination and $Ca(OH)_2$ addition to increase the water's pH level from approximately 6–8, water is finally stored prior to being used.

3. Methodology

3.1. Water sampling procedure

In order to characterise the chemical and microbiological quality of the water, six sampling points were selected for this investigation, and new data were generated from two field campaigns carried out in February and June, 2011.

Water was sampled from two wells, W1 and W2, two springs, S1 and S2, and a regulation pond, P (Fig. 1). Furthermore, rejected brine samples (data not shown here) from the desalination plant were analysed as an extra control point of the process. During the field campaigns, groundwater level measurements as well as *in situ* pH, EC, alkalinity, ALK, and temperature measurements were carried out at each sampling point with an Eijkelkamp® 18.28 multiparameter recording device.

A set of five water samples were collected at each sampled point for the following determinations: physico-chemical parameters, major ions, emerging contaminants-priority substances, heavy metals and microbiologic analysis. Water samples for physico-chemical and microbiological determinations were stored in 0.5 L PE-LD bottles. For emerging contaminants including surfactants, water samples were stored in 1 L amber glass bottles with Teflon coated cap. The surfactant sample set was stabilised with formaldehyde (4%) to prevent biological degradation. The third set of samples were acidified *in situ* with 3% HNO_3, filtered through a 0.45 μm membrane

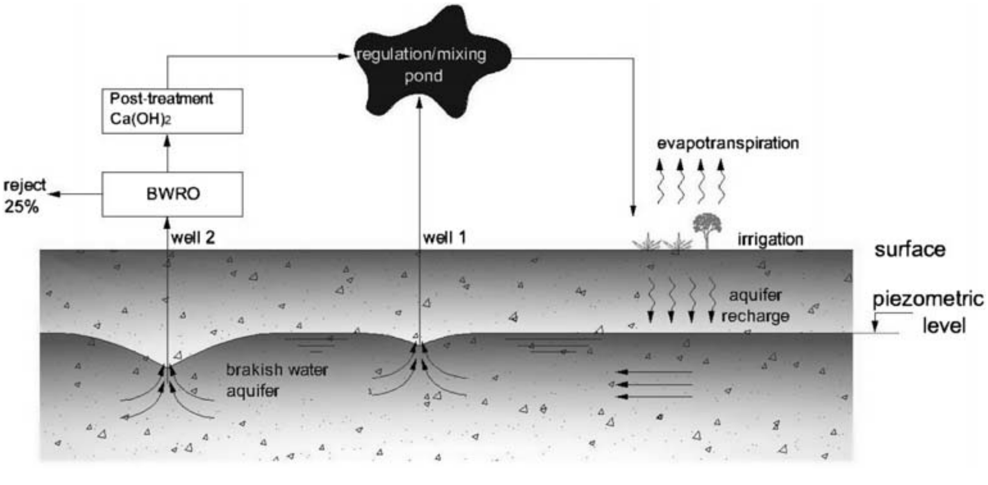

Fig. 2. Conceptual scheme of the desalination system.

(Millipore®) and stored in PE-LD bottles for heavy metals analysis. All samples were immediately stored in an insulated container chilled with ice and sent to the laboratory for analysis. Transit time for sample shipment was less than 24 h.

3.2. Chemical compounds and analytical techniques

Major ions and physico-chemical parameters were determined at the IMDEA-AGUA laboratory following standard procedures. Chemical analyses displayed acceptable ion balance errors.

Microbiological analysis included coliform and *Escherichia coli* determinations by the Colilert® 18 h method at the department of genetics and microbiology of the autonomous university of Barcelona.

A total of 209 emerging pollutants grouped into the following classes were analysed: 125 pharmaceutical compounds (Phs), 20 polyaromatic hydrocarbons, PAHs and dioxins, 46 pesticides, three volatile priority pollutants as well as the most commonly used anionic surfactants (Table 1).

The selection of compounds from the different classes was based on the following:

The metabolic routes of Phs, their common use as household products, their inclusion as a priority substance as defined by the 2008/105/EC Directive addressing environmental quality and literature search. Selected surfactants, the linear alkylbenzene sulfonate (LAS) included four homologues (C10, C11, C12, and C13) and their degradation products sulfophenylcarboxylate acids, and SPCs.

The selection of heavy metals included Cu, Cd, Pb, Hg, Ni, Zn, Sn, Pt, Pd and Tl.

Emerging compounds and heavy metal determinations were carried out at the department of physical and analytical chemistry at the university of Jaen. Heavy metal concentrations were determined by inductively coupled plasma mass spectrometry (ICP-MS). A synthesis of other applied techniques is reported in Table 2. Analysis of surfactants was provided by the Department of Physical-Chemistry at the University of Cadiz. High performance liquid chromatography–mass spectrometry was used to identify and quantify target compounds, based on the analytical protocol developed by Lara-Martín et al. [29].

The state of saturation (saturation index (SI)) of groundwater samples and mixed water samples for relevant minerals and the ionic speciation were calculated with the PHREEQC code [30]. The applied thermodynamic data base was minteq.v4.dat [31]. Often, no equilibrium is reached, and the SI state indicates the direction in which the process may go; for subsaturation dissolution is expected whilst supersaturation suggests precipitation. Speciation and equilibrium calculations with respect to mineral phases were carried out for groundwater (W1, W2, S1, S2) and pond samples (P) for both sampling campaigns.

4. Results and discussion

4.1. Major ions and physico-chemical parameters

A summary of the physico-chemical characteristics and major ions content for the different waters sampled (groundwater from wells and springs and pond water (P)) and sampling campaigns is presented in Table 3.

All water samples are characterised by high EC values (between 1,355 and 8,500 $\mu S\,cm^{-1}$), where SO_4^+, Cl^- and Na^+ are the most dominant ions originating from the evaporitic geologic layers. Although groundwater composition is quite homogeneous, the S1 sampling point which is the seepage of the slow regional system outflow shows the highest concentrations of these ions. Groundwater samples show neutral pH ≈ 7, whereas P are more basic with pH > 8 due to the desalination process. Seasonal physico-chemical groundwater changes were not observed in wells and spring samples. However, for pond samples (P), a 40% increase in TDS is detected in summer with respect to winter, which can be related to the greater groundwater fraction mixed in the pond, 20% in summer, as well as due to an enrichment of major ions in stored water due to evaporation. The sodium adsorption ratio, SAR, and EC values for P (Table 3) applied for landscape irrigation, indicate a low to moderate soil sodicity hazard.

Results of water sample saturation indices estimated from hydrochemical modelling with the PHREEQC code show that water is subsaturated with respect the majority of all possible mineral phases at the pH range existing in the study area. At neutral pH, groundwater presents a sodium sulphate–chloride facies and is in equilibrium, i.e. SI ≈ 0, with respect to anhydrite, $CaSO_4$, aragonite, $CaCO_3$, calcite $CaCO_3$, dolomite, $CaMg(CO_3)_2$, gypsum, $2H_2O{\cdot}CaSO_4$, fluorite, CaF_2 and magnesite, $MgCO_3$. Water from S2 samples are slightly supersaturated, i.e. SI > 0, with respect to aragonite, calcite and dolomite. As pH increases in mixed samples with pH >8, the water samples remain in equilibrium with the aforementioned minerals as well as with Cu, Smithsonite $ZnCO_3$, and ZnO. The increase of SI values observed in the summer campaign should be highlighted as they are supersaturated, i.e. SI > 0 with respect to Zn oxy-hydroxides

Table 1
List of micro-contaminants analysed classified according to this class group

Phs
11-Nor-9-carboxy-THC
3-Methylxanthine
4-Amino-antipiryne (4-AA)
Acetylmorphine
Amphetamine
Antipyrine
Atenolol
Atropine
Bendroflumethiazide
Benzafibrate
Benzalconium chloride total
Benzothiazol
Benzoylecgonine
Caffeine
Cannabidol
Carbadox
Carbamazepine
Cefotaxime
Cimetidine
Cis-diltiazem
Clarithromycin
Clembuterol
Cloxacillin
Cocaine
Codeine
Compactin (Mevastatin)
Cotinine
Danofloxacin
delta-9-THC
Demeclocycline
Dicloxacillin
Digoxigenin
Digoxin
Diphenhydramine
Doxycyclyne
EDDP
Enalapril
Enoxacin
Enrofloxacin
Ephedrine
Erythromycin
Estrona
Ethion
Ethoxyquin
Ethylamphetamine
Ethylmorphine
Famotidine
Fenofibrate
Indomethacine
Josamycin
Ketamine
Ketoprofen
Leucomalachite green
Lincomycin
Lomefloxacin
Malachite green
MDA
MDEA
MDMA
Mebendazole
Meclofenamic acid
Mefenamic acid
Metformin
Methadone
Methamphetamine
Metronidazole
Miconazole
Minocycline
Morphine
Naproxen
Nicotine
Nifurexazide
Norfloxacin
N-nitrosodiethylamine
N-nitrosodimethylamine
N-nitrosodi-n-dibutylamine
N-nitrosomethylethylamine
N-nitrosomorpholine
N-nitroso-n-diphenylamine
N-nitrosopiperidine
N-nitrosopyrrolidine
Ofloxacin
Oxacillin
Oxolinic acid
Oxytetracicline
Paracetamol (Acetaminophen)
Phenacethin
Phenylbuthazone
Phenylephrine
Pindolol
Pipemidic acid
Pravastatin
Propranolol
Propyphenazone
Ranitidine
Roxithromycin
Sulfamethizole
Sulfamethoxazole (SMZ)
Sulfanilamide
Sulfapyridine
Sulfathiazole
Triethyl phosphate
Theobromine
Theophylline
Thiabendazole
Timolol
Tributyl phoshate
Tributyltin chloride
Trimethoprim
Tylosine
Warfarin

PAHs and dioxins
Acenaphtylene
Anthracene
Benzo(a)anthracene
Benzo(a)pyrene
Benzo(b)fluoranthene
Benzo(ghi)perylene
Benzo(k)fluoranthene
Chrysene
Dibenzo(a,h)anthracene
Fenamiphos
Fenthion
Fluorene
Indene(1,2,3-cd)pyrene
Oxyfluorfen
Phenanthrene
Prometon
Prometryn
Pyrene
α-Cypermethrin
λ-cyhalothrin

Pesticides
4,4´-DDE
4,4´-DDT
Alachlor
Aldrin
Alfa-hexachlorocyclohexane
Alpha-endosulfan
Ametryn
Atrazin desethyl
Atrazine
Chlorotetracycline
Chlorotoluron
Delta-hexachlorohexane
Deltametrin
Desethyl terbuthylazine
Diazinon
Dieldrin
Diuron
Endosulfan sulphate
Endrin
Ethion
Lindane
Heptachlor
Hexachloro1,3-butadiene
Hexachlorobenzene
Imidacloprid
Iprodion
Isodrin
Isoproturon
Metoxychlor
Parathion ethyl
Parathion methyl
Pentachlorobenzene
Procymidone
Propazine
Pyrimiphos methyl
Simazine
Terbutrin
Terbutylazine
Trifluralin

Volatile priority pollutants
1,2,3-Trichlorobenzene (TCB)
1,2,4-TCB
1,3,5-TCB

Metals
Cd
Cu
Hg
Ni
Pb
Pd
Pt
Sn
Tl
Zn

(*Continued*)

Table 1 (*Continued*)

Phs			
Flufenamic acid	Salbutamol	Beta-endosulfan	*Surfactants*
Flumenique	Sarafloxacin	Beta-hexachlorocyclohexane	LAS-10
Gemfibrozil	Spiramycin I	Buprofezin	LAS-11
Heroin	Sulfachloropyridazine	Carfentrazone ethyl	LAS-12
Hydroflumethiazide	Sulfadiazine	ChlorfenvinphosA	LAS-13
Hydroflumethiazide	Sulfadimethoxin	ChlorfenvinphosB	SPC
Ibuprofen	Sulfamerazine	Chloropyrifos ethyl	

and carbonates, Ca and Mg carbonates and Cu, indicating that mineral precipitation can occur.

4.2. Emerging and priority pollutants

Emerging compounds detected in water samples, according to the group classes, CAS number and main uses are presented in Table 4. Many of the selected target compounds were below their individual limit of detection, LOD, see Table 1. Only 11 out of 125 Phs, 9 out of 20 PAHs and dioxins, 19 out of 46 pesticides and four LAS homologues were found in water samples at varying average concentrations. Volatile priority pollutants were not detected in either of the sampling campaigns. Due to the ubiquity and level of LAS concentration (μg L^{-1}), this group will be independently discussed.

Analytical results of detected emerging contaminants in water samples are summarised in Table 5. Significant differences in concentrations are observed among the analysed water types, ranging from non-detected compounds, or below LOD, to 2,950 ng L^{-1} for the individual micro-contaminant theobromine, except for LAS with concentrations in the order of μg L^{-1}.

According to the number of detected compounds, pesticides are the most important group, followed by Phs, in both the February and June campaigns. When considering the mass of compounds, significant changes are observed between February and June. In February, pesticides account for 69% of the total mass of emerging contaminants detected; in June, Phs were the most important group, accounting for 91% of the total mass. This fact is related to the extreme concentration of theobromine, theophylline and caffeine at the S1 seepage sampling point. Finally, PAHs and dioxins which only represent 4% in February and 2% in June of the total mass are the group of compounds with the lowest frequency of detection. This fact is related to the intensive human occupation of this area, both for agricultural uses and urban settlements.

As presented in Table 5, only 11 out of 125 Phs compounds were detected. Almost all of them were present at a concentration below 0.1 μg L^{-1}, which is the threshold value considered in this investigation due to the absence of existing regulations. Caffeine, nicotine, sulfanilamide, theobromine and theophylline have the highest concentrations in groundwater samples. Theobromine also was detected with an elevated concentration in P samples. It is worth noting that a high nicotine concentration, a common compound in many pesticides, was found in sampling point S1 which is located in an agricultural area. Theobromine and theophyline, both broncho-and-vaso dilators and caffeine, common active ingredients in some Phs, were the compounds detected with the highest concentrations in the S1 sampling location. Their presence could be a result of uncontrolled spills.

Among the group of PAHs, fluorene, oxifluorfen, phenanthrene and pyrene were detected in all sampling points at ng L^{-1} concentrations. Dioxines were not detected.

Pesticides also occurred frequently and are the group with the greatest number of different detected compounds. The highest pesticide concentration in water samples was detected in February when common agricultural practices of the area include agrichemical applications. Pyrimiphos methyl (acaricide and insecticide) was the compound with the highest concentration (935 ng L^{-1}); followed by carfentrazone ethyl and desethyl terbuthylazine.

LAS levels were unusually high (~110 μg L^{-1}), and the LAS homologue percentage distribution (C10LAS, C11LAS, C12LAS and C13LAS) is closer to those commonly found in commercial mixtures. Similar concentration values in aquatic ecosystems have been reported in other studies [32]. LAS presence in groundwater could be originated from untreated wastewater that is infiltrated to the aquifer.

Regarding target compounds included in the 2008/105/EC Directive, only α-endosulfan and Ni were found above the maximum allowable concentra-

Table 2
Analytical methods applied for the chemical determination of the analysed compounds. BWRO: Brackish Water RO

Compounds	Sample Pre-treatment	Extraction method	Analytical method
Group I			
Phs: 11-nor-9-carboxy-THC, 3-Methylxanthine, 4-AA, Acetylmorphine, Amphetamine, Antipyrine, Atenolol, Atropine, Bendroflumethiazide, Benzafibrate, Benzalconium, Chloride total, Benzothiazol, Benzoylecgonine, Caffeine, Cannabidol, Carbadox, Carbamazepine, Cefotaxime, Cimetidine, Cis-diltiazem, Clarithromycin, Clembuterol, Cloxacillin, Cocaine, Codeine, Compactin (Mevastatin), Cotinine, Danofloxacin, delta-9-THC, Demeclocycline, Dicloxacillin, Digoxigenin, Digoxin, Diphenhydramine, Doxycyclyne, EDDP, Enalapril, Enoxacin, Enrofloxacin, Ephedrine, Erythromycin, Estrona, Ethion, Ethoxyquin, Ethylamphetamine, Ethylmorphine, Famotidine, Fenofibrate, Flufenamic acid, Flumenique, Gemfibrozil, Heroin, Hydroflumethiazide, Hydroflumethiazide, Ibuprofen, Indomethacine, Josamycin, Ketamine, Ketoprofen, Leucomalachite green, Lincomycin, Lomefloxacin, Malachite green, MDA, MDEA, MDMA, Mebendazole, Meclofenamic acid, Mefenamic acid, Metformin, Methadone, Methamphetamine, Metronidazole, Miconazole, Minocycline, Morphine, Naproxen, Nicotine, Nifurexazide, Norfloxacin, N-nitrosodiethylamine, N-nitrosodimethylamine, N-nitrosodi-n-dibutylamine, N-, itrosomethylethylamine, N-nitrosomorpholine, N-nitroso-n-diphenylamine, N-nitrosopiperidine, N-nitrosopyrrolidine, Ofloxacin, Oxacillin, Oxolinic acid, Oxytetracicline, Paracetamol, Acetaminophen), Phenacethin, Phenylbuthazone, Phenylephrine, Pindolol, Pipemidic acid, Pravastatin, Propranolol, Propyphenazone, Ranitidine, Roxithromycin, Salbutamol, Sarafloxacin, Spiramycin I, Sulfachloropyridazine, Sulfadiazine, Sulfadimethoxin, Sulfamerazine, Sulfamethizole, SMZ, Sulfanilamide, Sulfapyridine, Sulfathiazole, TEP (Triethyl phosphate), Theobromine, Theophylline, Thiabendazole, Timolol, Tributyl phosphate, Tributyltin chloride, Trimethoprim, Tylosine, Warfarin	Filtration (0.7 μm) Glass-fiber filters) pH adjustment (pH 8)	Solid phase extraction (Oasis® HLB, 200 mg, 6 cm^3) MeOH as solvent	Liquid chromatography-Q-TRAP • Mass spectrometry • *Column:* C-18 (3 × 250 mm, 5 μm particle size) • *Interfase polarity:* ESI (+) y ESI (−) • *Mobile phase:* ESI (+), water (formic acid 0.1%)/ACN ESI (−), water (0.05% ammonium formate)/ACN • *Operation mode:* MRM
Pesticides: Alachlor, Ametryn, Atrazine, ChlorfenvinphosA, ChlorfenvinphosB, Chloropyrifos ethyl, Chlorotoluron, Deltametrin, Diazinon, Diuron, Iprodion, Isoproturon, Parathion ethyl, Parathion methyl, Procymidone, Propazine, Simazine, Terbutrin, Buprofezin, Carfentrazone ethyl, Desethyl, erbuthylazine, Imidacloprid, Pyrimiphos methyl			
Group II			
PAH and dioxines: Acenaphtylene, Anthracene, Benzo(a)anthracene, Benzo(a)pyrene, Benzo(b)fluoranthene, Benzo(ghi)perylene, Benzo(k)fluoranthene, Chrysene, Dibenzo(a,h)anthracene, Fenamiphos, Fenthion, Fluorene, Indene(1,2,3-cd)pyrene, Oxyfluorfen, Phenanthrene, Prometon, Prometryn, Pyrene, α-Cypermethrin, λ-cyhalothrin	No Filtration pH adjustment (pH 3) NaCl Addition	Liquid–liquid extraction. Hexane as solvent	Gas chromatography • Mass spectrometry/mass spectrometry • *Column:* HP-5 ms (5% diphenyl 95% dimethylsiloxane) • *Operation mode:* MS or MS/MS
Pesticides: 4,4´-DDE (Dichlorodiphenyldichloroethylene), 4,4´-DDT (dichlorodiphenyltrichloroethane), Aldrin, Alpha-endosulfan, Ametryn, Atrazine, Atrazin desethyl, Beta-endosulfan, ChlorfenvinphosA, ChlorfenvinphosB, Chloropyrifos ethyl, Chlorotetracycline, Chlorotoluron, Deltametrin, Diazinon, Dieldrin, Diuron, Endosulfan sulphate, Endrin, Ethion, Heptachlor, Imidacloprid, Iprodion, Isodrin, Isoproturon, Metoxychlor, Parathion ethyl, Parathion methyl, Procymidone, Propazine, Simazine, Terbutrin, Terbutylazine, Trifluralin			
Group III			
Pesticides: Alachlor, Alfa-hexachlorocyclohexane, Beta-hexachlorocyclohexane, Delta-hexachlorohexane, Gamma-hexachlorocyclohexane(lindane), Hexachloro1,3-butadiene, Hexachlorobenzene, Pentachlorobenzene	No Filtration pH adjustment (pH 3) NaCl addition	Liquid–liquid extraction Hexane as solvent.	Gas chromatography–Mass spectrometry/mass spectrometry–*Column:* HP-5 ms (5% diphenyl 95% dimethylsiloxane)–*Operation mode:* MS or MS/MS
Volatile prioritary polutants: 1,2,3- TCB, 1,2,4- TCB, 1,3,5- TCB			
Group IV			
Heavy metals: Cadmium, Cupper Lead, Nickel, Mercury, Palladium, Platinum, Tin, Thallium and Zinc	Filtration (0.7 μm Glass–fiber filters) 1:2 dilution with 3% HNO_3	None	(ICP-MS) • *RF power:* 1,500 W • *Plasma gas flow:* 15 L/min • *Nebulizer gas flow:* 1 L/min • *Auxiliary gas flow:* 0.9 L/min • *Sample uptake rate:* 0.25 mL/min

Table 3
Physico-chemical parameters and major ion content for the February and June sampling campaigns

Sample sites	W1	W2	S1	S2	P	W1	W2	S1	S2	P
	11/February/11					7/June/11				
Physico-chemical charact.										
pH	7.0	7.0	7.0	7.8	8.2	7.1	7.0	7.0	7.6	8.6
T (℃)	19.7	20.5	18.9	14.2	13.7	20.8	21.3	19.5	18.2	21.5
Eh (mV)	87	89	89	42	21	89	93	87	59	3
EC ($\mu S\,cm^{-1}$)	8,010	7,080	8,500	7,070	1,355	7,860	6,990	8,210	7,030	1,994
Hardness ($mg\,L^{-1}$ $CaCO_3$)	1,939.4	1,693.8	2,603.4	1,685.3	248.9	1,965.3	1,733.2	2,657.5	1,697.2	403.8
Alk TAC ($mg\ L^{-1}$ $CaCO_3$)	279.6	270.2	277.1	249.6	74.6	279.5	267.3	276.8	254.7	96.7
SAR	11.2	10.6	9.0	10.5	5.5	10.9	10.5	8.9	10.3	5.8
Major ions ($mg\,L^{-1}$)										
Na^+	1,129.8	999.4	1,056.8	986.6	198.9	1112.8	1,002.5	1,055.9	975.5	267.6
K^+	24.9	22.1	23.3	13.3	4.5	24.9	21.7	26.7	14.7	6.6
Ca^{2+}	378.4	309.1	540.1	329.6	54.5	383.2	322.9	553.7	332.2	83.4
Mg^{2+}	241.5	223.9	304.7	209.4	27.4	244.9	225.1	309.6	210.7	47.5
F^-	1.5	1.9	1.0	1.6	0.2	1.9	2.3	1.7	2.2	2.8
Cl^-	1,654.9	1,266.5	1,655.9	1,423.4	263.4	1,635.9	1,290.2	1,659.1	1,430.1	373.0
NO_3^-	113.2	132.9	102.1	64.7	47.4	117.9	137.9	106.1	65.6	42.9
SO_4^{2-}	1,689.5	1,679.4	2,173.1	1,526.0	194.6	1,713.9	1,727.4	2,228.2	1,558.7	327.6
HCO_3^-	341.1	329.7	338.1	304.5	90.9	340.9	326.1	337.6	310.8	110.1
CO_3^{2-}	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	3.9

Note: W1, W2: wells 1 and 2; S1, S2: springs; P: pond.
T = Temperature; EC = electrical conductivity; Eh = redox potential; Alk TAC = total alkalinity; SAR = sodium adsorption ratio.

tion defined by this Directive. A concentration of α-endosulfan of 6.2 $ng\,L^{-1}$ was detected in the S2 sampling point in the February sampling campaign, where the maximum accepted level for both α- and β-endosulfan is 4 $ng\,L^{-1}$. The Ni heavy metal was detected in all groundwater sampling points with an average concentration of 33.54 $\mu g\,L^{-1}$.

The total concentration of detected compounds in groundwater samples (W1, W2, S1, S2) and its variability are indicative of the aquifer background. The detected concentration in sample site P is similar to the aquifer background levels, and only slight changes are observed. It appears to be that the RO treatment removal efficiency is low for these types of compounds. This fact is in agreement with previous studies [33]. However, LAS values in P point samples (mixed water) is one order of magnitude lower than the rest of sampled points, as most of the surfactants have been already removed after desalination.

4.3. Heavy metals

Among the heavy metals, only Cu, Ni, Pb and Zn were detected in all water samples. According to the *SI* results, water is subsaturated with respect to mineral species containing the aforementioned metals, and dissolution is expected. Ni concentration ranged between 44.4 and 22.0 $\mu g\,L^{-1}$ in groundwater samples and decreases to an average of 8.8 $\mu g\,L^{-1}$ for mixed water. The observed decrease is also related to the physico-chemical parameters for P water (pH >8 and low Eh). Detected Zn concentrations are always greater than 250 $\mu g\,L^{-1}$ in all samples, and a small variability in the groundwater samples indicates a geochemical source for this compound. Similar results are found in the literature [34]. Changes of Ni and Zn concentrations in pond samples may be the consequence of organic matter complexation, and physico-chemical characteristics of water such as pH and Eh, water mixing; however, confirming this would require further investigation. Also, a possible origin from industrial wastes should not be discarded.

4.4. Microbiology

From a microbiological point of view, 461 and 410 cfu per 100 mL of *E. coli* in the P samples were detected in water samples collected in February and June, respectively. The concentration of total coliform bacteria was 648×10^4–3.65×10^4 cfu per 100 mL in the

Table 4
CAS number and type of application for the detected compounds

	CAS number	Application
Phs ($\sim$ng L^{-1})		
Antipyrine	60-80-0	Analgesic and antipyretic
Caffeine	58-08-2	Central nervous system stimulant
Carbamazepine	298-46-4	Anticonvulsant
Cotinine	486-56-6	Nicotine derivate
Nicotine	54-11-5	Tobacco ingredient, pesticide
Phenylephrine	59-42.7	Decongestant, pupil dilator, blood pressure agent
SMZ	723-46-6	Bacteriostatic antibacterial
Sulfanilamide	63-74-1	Sulfonamide antibacterial
Theobromine	83-67-0	Bronchodilator and vasodilator
Theophylline	58-55-9	Diuretic, muscle relaxant, bronchial dilation,
PAHs and dioxines ($\sim$ng L^{-1})		
Acenaphtylene	208-96-8	Dye synthesis, insecticides, fungicides, plastic industry
Anthracene*	120-12-7	Source of dyestuffs
Benzo(a)anthracene*	55-56-3	May be expected to be carcinogen
Benzo(ghi)perylene*	191-24-2	Byproduct of fuel burning
Chrysene	218-01-9	Manufacture of dyes
Fluorene	86-73-7	As a luminophore in organic light-emitting diodes.
Oxyfluorfen	42874-03-3	Herbicide
Phenanthrene	85-01-8	Cigarette smoke; irritant, photosensitising skin to light
Pyrene	129-00-0	Dyes and dye precursors
Pesticides ($\sim$ng L^{-1})		
4.4´-DDE	72-55-9	Pesticide
4,4´-DDT*	50-29-3	Pesticide
Endosulfan*	115-29-7	Cyclodiene insecticides. One of two components of endosulfan (115-29-7)
Atrazine*	1912-24-9	Herbicide
Buprofezin	953030-84.7	Insecticide, chitin synthesis inhibitors
Carfentrazone ethyl	128,639-02-1	Herbicide (Triazolone herbicides)
Chloropyrifos ethyl*	2,921-88-2	Acaricide, insecticide and nematicide
Desethyl terbuthylazine	30,125-63-4	Pesticide
Diuron*	330-54-1	Herbicide
Endosulfan sulphate*	1,031-07-8	Acaricide, insecticide
Hexachlorobenzene*	118-74-1	Fungicide, as a seed treatment, to control fungal disease bunt
Pentachlorobenzene*	608-93-5	Fungicide
Procymidone	32,809-16-8	Fungicide
Pyrimiphos methyl	29,232-93-7	Acaricide, insecticide
Simazine*	122-34-9	Herbicide of the Triazine class, to control broad-leaved weeds
Terbutylazine	5,915-41-3	Herbicide (Chlorotriazine herbicides)
Surfactants ($\sim\mu$g L^{-1})		
LAS C10	1,322-98-1	Detergent
LAS C11	68,411-30-3	Detergent
LAS C12	69,669-44-9	Detergent
LAS C13	68,411-30-3	Detergent

Note: *included in 2008/105/EC Directive.

Table 5
Analytical results for emerging contaminants, priority compounds (defined in the 2008/105/EC Directive), and heavy metals

Sampled points	W1	W2	S1	S2	P	W1	W2	S1	S2	P
	11/February/11					7/June/11				
Phs (ng L^{-1})										
Antipyrine	–	7.70	–	–	–	–	–	–	–	–
Benzalconium chloride total	–	–	–	–	–	–	298.80	62.10	33.40	42.60
Caffeine	–	–	132.20	50.00	69.60	–	–	654.00	–	34.60
Carbamazepine	13.90	45.20	–	–	–	–	50.50	–	–	–
Cotinine	5.00	7.90	22.70	10.30	22.80	–	11.40	109.00	–	–
Nicotine	–	–	265.10	–	–	–	–	–	–	–
Pindolol	–	–	–	–	–	21.80	–	–	–	–
SMZ	35.10	39.90	23.00	–	–	–	–	–	–	–
Sulfanilamide	100.40	136.90	42.70	–	–	–	113.30	38.70	–	–
Theobromine	–	–	208.90	–	599.80	–	–	2,950	–	–
Theophylline	–	–	99.30	–	–	–	–	769.00	–	–
Total Phs	154.40	237.60	793.90	60.30	692.20	21.80	474.00	4582.8	33.40	77.20
Total Phs (%)			27					91		
PAHs and dioxines (ng L^{-1})										
Acenaphtylene	–	0.35	–	0.23	–	–	–	–	–	–
Anthracene*	85.84	–	3.61	–	–	4.83	–	–	–	–
Benzo(a)anthracene	1.53	–	–	–	0.50	–	–	–	–	–
Benzo(ghi)perylene	–	–	–	–	–	–	0.23	–	–	–
Chrysene	1.90	0.96	–	0.83	–	0.63	0.95	–	0.80	–
Fluorene	2.42	2.32	1.02	1.37	0.88	1.11	1.28	0.71	1.00	0.71
Oxyfluorfen	2.69	39.54	4.72	1.88	1.25	1.11	2.63	4.80	1.83	1.49
Phenanthrene	15.27	16.80	6.75	10.42	5.10	9.37	13.01	6.20	9.24	5.09
Pyrene	24.67	9.99	5.15	8.59	3.72	9.13	12.03	4.53	8.72	5.13
Total PAHs and dioxines	134.32	69.96	21.25	23.32	11.45	26.18	30.13	16.24	21.59	12.42
Total PAHs and dioxines (%)			4					2		
Pesticides (ng L^{-1})										
4.4´-DDE	–	–	–	–	–	–	–	–	0.56	–
4.4´-DDT*	–	0.38	–	–	–	–	–	–	0.29	–
α-Endosulfan*	–	–	–	6.20	-	–	–	–	–	–
Ametryn	–	–	–	–	–	–	37.30	–	–	–

(*Continued*)

Table 5 (*continued*)

Sampled points	W1	W2	S1	S2	P	W1	W2	S1	S2	P
	11/February/11					7/June/11				
Atrazine*	–	–	–	–	–	9.40	6.50	5.30	–	8.80
Atrazin desethyl	–	–	–	–	–	20.10	16.70	–	6.50	–
Buprofezin	14.20	13.90	–	–	16.30	–	–	–	–	–
Carfentrazone ethyl	114.70	94.90	105.90	76.10	93.50	–	–	–	–	–
Chloropyrifos ethyl	3.72	7.80	1.86	3.75	2.38	9.18	15.53	4.61	6.63	5.07
Desethyl terbuthylazine	25.00	19.90	5.20	7.30	–	34.10	32.10	–	–	–
Diuron*	–	–	–	–	–	1.11	–	–	–	–
Endosulfan sulphate*	–	–	3.12	–	–	–	1.21	6.31	–	–
Hexachlorobenzene*	1.44	7.08	1.52	1.43	0.39	1.58	11.73	1.58	1.72	0.65
Pentachlorobenzene*	–	0.24	–	–	–	–	–	–	–	–
Procymidone	–	–	–	–	–	1.52	–	–	–	–
Propazine	–	–	–	–	–	–	–	24.90	24.70	25.70
Pyrimiphos methyl	935.40	784.60	812.30	899.10	779.40	–	–	–	–	–
Simazine*	11.10	11.50	2.50	2.70	2.30	18.40	21.60	–	–	–
Terbutylazine	0.92	0.58	0.22	0.38	–	4.18	4.55	1.80	2.01	2.37
Total pesticides	1,106.48	940.88	932.62	996.96	894.27	99.57	147.22	44.50	42.41	42.59
Total pesticides (%)			69					7		
Phs + PAHs + Pesticides	1,395.20	1,248.44	1,747.77	1,080.58	1,597.92	147.55	651.35	4,643.54	97.40	132.21
Surfactants (μg L^{-1})										
LAS C10	19.20	13.30	4.20	12.30	3.70	16.00	8.60	1.70	0.50	1.00
LAS C11	86.00	34.10	5.70	47.00	6.60	83.40	60.90	17.70	12.40	14.00
LAS C12	47.60	23.60	5.00	27.20	3.60	34.80	15.70	3.40	3.50	2.80
LAS C13	66.60	28.20	3.00	28.80	2.40	16.60	7.70	5.50	1.40	2.10
Total LAS	219.40	99.20	17.90	115.30	16.30	150.80	92.90	28.30	17.70	19.90
Metals (μg L^{-1})										
Cu	6.95	6.65	3.40	2.80	4.65	10.70	4.30	4.50	3.80	5.10
Ni*	30.85	27.95	44.35	31.00	8.80	27.30	22.00	36.10	26.10	9.70
Pb*	3.95	3.70	3.40	3.45	3.65	5.10	4.20	4.30	4.40	4.50
Zn	270.15	311.10	289.15	280.95	374.90	257.60	261.40	296.10	301.40	359.70

Note: W1, W2: wells 1 and 2; S1, S2: springs; P: pond.– under LOD; * included in 2008/105/EC Directive.

February and June campaigns, respectively. *E. coli* values do not meet the water quality standards indicated in the Royal Decree R.D. 1620/2007 which regulates reclaimed water used for urban garden irrigation. The pond is the habitat of wildlife, among them there is a colony of ducks, which contributes to the presence of the microbiological contamination. Changes in concentration between February and June can be explained by the different biological activity that takes place in winter and summer seasons.

5. Conclusions

The desalination of groundwater is generally perceived as a safe water resource, especially in arid areas. However, groundwater may contain high levels of chemicals which may put ecosystems at risk and need to be removed before use.

This study shows that only a small number of the monitored active compounds were detected in sampled waters. Phs showed the greatest variability which is most likely associated to consumption pattern, whilst pesticides were the most frequently detected chemicals at about 34% of the total monitored compounds, probably due to the high fraction of the agricultural land cover in the study area. The effect of seasonality within this data may be the result of agricultural practices as observed though the different presence of pesticides.

It is important to note that surfactants LAS, PAHs and dioxins were detected in most of the water samples; this indicates the presence of wastewater effluents of urban origin. The ubiquitous presence of LAS is related to the low-sorption capacity and its wide variety of applications, ranging from cleaning products to pesticide formulation (among others). They are very good markers of sewage-derived pollution.

The concentration of detected compounds met drinking water standards for the specified parameters, except for the microbiological quality of the irrigation water. In regards to the EC Directive on priority substances list, only endosulfan in one out of six sample points, and Ni in five out of six sample points, reported concentrations above the maximum allowable concentration.

It is important to note the state of saturation of blended water samples from the pond which is supersaturated with respect to carbonates. Pond water is used for landscape irrigation during the summer period. Changes in soil and aquifer porosity could occur due to the precipitation or dissolution of calcite. The results obtained have a significant implication for irrigation management, in terms that irrigation with water that presents high SI values could produce changes in soil structure. Also, it was detected that SAR index were slightly high so related problems, as clay swelling and dispersion, could appear.

The study also revealed that the removal of target compounds by desalination treatment does not show significant variations and also that the total concentration of chemicals in mixed water is generally similar to the rest of sampled points. Based on the results obtained, anticipation of future impacts due to the presence of emerging contaminants potentially causing ecosystem harm, as well as the effect of in-fill soil and aquifer material pore spaces by irrigation water is required.

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