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1 **Removal of 39 contaminants of emerging concern found in wastewater effluent by coupling**  
2 **nanofiltration and infiltration into saturated soil column.**

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13 Highlights:

14 Nanofiltration using NF-270 has shown interesting removal for all parameters studied.  
15 Soil infiltration mainly retains or slow down positive compounds due to soil charge.  
16 Nanofiltration permeate was used for the first time coupling with soil infiltration.  
17 Contaminants of emerging concern are removed at 92% with coupling treatment.  
18 22 out of the 39 compounds found in effluent were not detected after coupling.

19

20 **Abstract:**

21 Management of water is evolving, due to water scarcity, and the reuse of wastewater  
22 treatment plant effluent is more and more investigated. New applications of the reuse such as  
23 the refilling of aquifer could be investigated. Soil aquifer treatment (SAT), sometimes used as  
24 natural advanced treatment, has shown some limitations for the removal of several  
25 contaminants of emerging concern (CECs). In this study, the investigation of a coupling  
26 advanced treatment process using nanofiltration followed by soil infiltration has been  
27 performed. The aim of the study was focused on the removal efficiency of the coupling  
28 treatment for 39 CECs. The CECs studied were detected in the wastewater treatment plant  
29 effluent and removal efficiencies were determined based on the natural concentrations of  
30 CECs without additional spiking. The coupling has shown interesting results allowing to  
31 increase the performances of the treatments used alone. While nanofiltration alone leads to  
32 a decrease of 80% of the total concentration of CECs adding soil infiltration increases the  
33 removal to 92%. 22 CECs out of the 39, detected in the effluent, are not detected after the  
34 coupling advanced treatment. However, few CECs such as carbamazepine, irbesartan or  
35 hydrochlorothiazide have shown limited removal despite the use of combined treatment.  
36 **Keywords:** Pharmaceutical, Advanced treatment process, Soil infiltration, Removal efficiency,  
37 Wastewater treatment plant effluent, LC-MS/MS.

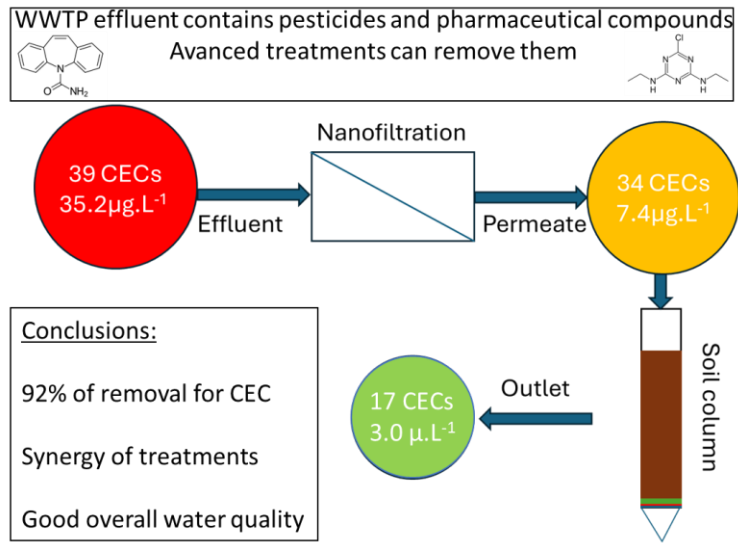
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41 Graphical abstract:

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45 **1.INTRODUCTION:**

46 Water is a fundamental resource for human being and their development. Population growth  
47 and urbanisation tend to accelerate the consumption of water (Cazcaro *et al.*, 2013; Tan *et al.*,  
48 2021). Coupling with climate change, more and more countries have to face water scarcity  
49 (Gosling and Arnell, 2016; Schewe *et al.*, 2014). Some countries or landscapes faced this  
50 problematic since several decades such as Israel, South of Spain, Australia, California, *etc.* Thus,  
51 management of water resource and particularly aquifers has become a hot button issue in the  
52 world. Natural infiltration does not provide enough water to counterbalance water withdrawal  
53 from the aquifers (Ousrhire and Gafiri, 2022). Such a situation can create a lack of water  
54 (Aidary and Kazemi, 2014) which might lead to the contamination of groundwater due to  
55 seawater intrusion in the aquifer if the level of groundwater becomes too low (Hajji *et al.*,  
56 2022). This kind of contamination is difficult to overcome but can be avoided by a smart  
57 management (Al-Yaqoubi *et al.*, 2021; Bachtouli and Comte, 2019). Different solutions could  
58 be used to control or limit groundwater uses. For instance, some countries use desalination in  
59 addition to groundwater input (Shahabi *et al.*, 2017). The desalination is performed thanks to  
60 several techniques such as thermal, electrochemical, filtration or ion exchange (Subramani and  
61 Jacangelo, 2015). Depending on the country, each technique can be suitable for providing  
62 additional water after balancing advantages and drawbacks (Castro *et al.*, 2020; Subramani  
63 and Jacangelo, 2015). Another important source of water come from reuse of wastewater  
64 treatment plants (WWTPs) effluents and can complete seawater and groundwater sources for  
65 some countries (Angelakis *et al.*, 2018). Indeed, countries used to water depletion have already  
66 widely develop the reuse such as Israel (Friedler *et al.*, 2006) while other countries are more  
67 and more interested by this source, supported by new legislations such as in Europe (Water  
68 reuse regulation (EU) 2020/741 and guidelines Minimum Requirements for water reuse,

69 2020). WWTPs effluents are of interest for several reasons: (i) its availability which is unlimited  
70 as long as WWTPs are operating, (ii) its spread over territories and (iii) its impact for some  
71 natural media which can be highly impacted by WWTPs effluents, particularly during dry  
72 period, in case of direct rejection (Lockmiller *et al.*, 2019; Rabiet *et al.*, 2005; Trommetter *et*  
73 *al.*, 2022). However, the last consideration might also be a brake for the reuse as surface water  
74 can lack during dry period. Quality of effluents also needs to be improved for being used  
75 directly (golf irrigation, public landscape irrigation, etc) or indirectly (aquifer recharge).  
76 Moreover, increasing effluents quality could help for social acceptance which still has to be  
77 meliorated even if a change of mind occurs since the last decades (Garin *et al.*, 2020; Rice *et*  
78 *al.*, 2016; Verhoest *et al.*, 2022). In case of wastewater reuse for potable use or irrigation of  
79 vegetables, one of the main fears of public opinion deals with the risk of contamination by  
80 some substances inducing a risk for health (Garin *et al.*, 2020; Rice *et al.*, 2016; Verhoest *et al.*,  
81 2022). Such a fear can be explained by the low attenuation of contaminants of emerging  
82 concerns (CECs), which are mainly pharmaceutical compounds, by WWTPs (Alexa *et al.*, 2022;  
83 Dolar *et al.*, 2012; Gabrielli *et al.*, 2023). However, CECs can be further eliminated with  
84 advanced treatments such as membrane filtration processes, advanced oxidation processes,  
85 granular activated carbon, ozonation, etc (Rizzo *et al.*, 2019). Even if these treatments are  
86 efficient enough to allow the use of wastewater for some activities it is necessary to combined  
87 multiple treatments to reach a very high-quality water to recharge aquifer. Very high-quality  
88 water could be a source of potable water after infiltration. Some studies have already been  
89 performed showing a good efficiency of coupling treatments such as short-soil aquifer  
90 treatment/nanofiltration (Cikurel *et al.*, 2006); O<sub>3</sub>/membrane filtration/biological methods (Qi  
91 *et al.*, 2010) or biofiltration/O<sub>3</sub>/ short-soil aquifer treatment (Zucker *et al.*, 2015).

92 In the present study, the coupling of nanofiltration followed by soil infiltration will be  
93 investigated for the first time to our knowledge with natural concentrations of CECs, from a  
94 few  $\text{ng.L}^{-1}$  to several  $\mu\text{g.L}^{-1}$ , found in effluent from a WWTP equipped with a membrane  
95 bioreactor (MBR). Our investigation proceeded in steps. Firstly, the performances of each  
96 treatment separately have been determined to optimize its efficiency alone with the selected  
97 wastewater effluent. Then, coupling treatment using the same effluent has been performed.  
98 Effluent was firstly treated by nanofiltration. Thus, the permeate recovered was used to feed  
99 a 30cm height saturated soil column for 24 days continuously. Performances of the different  
100 treatments were assessed by following some classical water parameters (chemical oxygen  
101 demand, physicochemical, ions) and by determining the concentration, along the treatments,  
102 of 39 CECs detected (out of 80 CECs quantifiable with an LC-MS/MS method previously  
103 developed (Sellier, 2023) in the WWTP effluent. Pristine concentrations of CECs were used for  
104 all experiments with no additional spiking. Finally, the results obtained for the coupling  
105 treatment will be discussed and compared with nanofiltration and soil infiltration alone and  
106 with other coupling treatments found in the literature to determine advantages and  
107 drawbacks of such a coupling.

108 **2. Materials and method:**

109 **2.1. Nanofiltration pilot:**

110 Nanofiltration was performed thanks to a pilot build by the European Institute for Membranes  
111 (Institut Européen des Membranes, Montpellier, France). The pilot is composed of 15L tank  
112 receiving the WWTP effluent, a nanofiltration module with a surface area of 144cm<sup>2</sup>; 2  
113 pressure sensors to determine the pressure of the feeding and the retentate (STS 127795,  
114 range 0-20 bars); a cooler Julabo 601F to control the temperature of the experiment (Julabo  
115 France SAS, Colmar, France); a 2.2kW pump to recirculate the water in the pilot (Barthod  
116 Pompe, Meyzieux, France); a balance PCE-TB series to follow the permeate flow (PCE  
117 Instruments, Soultz-sous-Forêt, France). Transmembrane pressure was adjusted between 0  
118 and 10 bars thanks to a GO regulator PR-1 series (Crane Instrumentation & Sampling PFT Corp.,  
119 Spartanburg, SC, USA)

120 The tangential velocity was fixed at 0.5m.s<sup>-1</sup> thanks to an alternator and the temperature was  
121 fixed at 20±1°C (Azaïs *et al.*, 2016). Two flat sheet membranes were used during the  
122 preliminary tests: NF-90, a « tight nanofiltration membrane », and NF-270 a « loose  
123 nanofiltration » membrane. Both Filmtec™ polyamide membranes were purchased from  
124 DuPont Water Solutions (Edina, MN, USA). Note that for the coupling experiment NF-270 was  
125 used. Before using, the membranes were soaked into ultrapure water for at least one night.  
126 Then a compacting was performed with a maximal pressure settled 2 bars above the working  
127 conditions. Pressure was increased by 2 bars increments (let 10 minutes for intermediate  
128 bearings and 1 hour for the maximal pressure). After each use, membranes were rinsed with  
129 ultrapure water and soaked into a 200mg.L<sup>-1</sup> NaNO<sub>3</sub> solution to prevent microbial  
130 development.

131 The pilot was cleaned with ultrapure water when performed in routine. Between two distinct  
132 experiments washing with 0.1M HCl solution (30 minutes at least), ultrapure water, 0.1M  
133 NaOH solution (30 minutes at least) and ultrapure water (until the conductivity was less than  
134  $10\mu\text{S}\cdot\text{cm}^{-1}$ ) was performed to avoid contamination.

135 Pilot was operated with a recirculation of the retentate into the feeding tank. Permeate was  
136 either collected at 60% and 80% conversion for preliminary studies or collected until 80%  
137 conversion (2days) and 90% conversion (2days) for the coupling experiment. The 32L collected  
138 over 4 operating days (2\*7.5L with 80% conversion and 2\*8.5L with 90% conversion) were  
139 homogenised, divided into bottles and frozen until their use to minimize the variation of the  
140 feeding solution over time (24 days). A scheme of the experimental setup is available in  
141 Appendix (Figure A1) for more details.

## 142 **2.2.Column setup:**

143 The infiltration in the soil column was carried out after nanofiltration. The 30cm-high column  
144 was built thanks to borosilicated glass portions. Each portion has an internal diameter of 10cm  
145 and a height of 12cm. The portions can be assembled thanks to O-ring seals and clamp rings.  
146 To avoid the fine soil particles running out the column, a metal grid with 0.5cm holes was put  
147 at the bottom of the column. The grid was covered with glass wool and a cleaned sand quartz  
148 layer (around 0.5cm). After each experiment, the column was dismantled, and each portion  
149 was cleaned with ethanol or acetone followed by a water cleaning.

150 The soil was collected in December 2022. Sampling site was located in Bezouze in France  
151 (X=819558; Y=6307953). Soil parameters are presented in Appendix (Table A1). Soil was  
152 compacted thanks to a 0.5kg mass launched from a 20cm height. The compacting was  
153 performed following ISO 21268-3:2019 recommendation (ISO 21268-3, 2019; Naka *et al.*,



154 2016). Soil columns were saturated before the experiments thanks to upflow saturation. The  
155 water used for the saturation was ultrapure water equilibrated with soil for one day, filtered  
156 at 0.45 $\mu$ m and finally introduced in column to flush it with volume corresponding to 3 pore  
157 volumes. Soils were naturally dried and sieved at 1mm.

158 Materials were chosen to minimize the loss of CECs by adsorption (glass, polypropylene or  
159 polytetrafluoroethylene (Demeau a)).

160 During the coupling experiment, the column of 30cm-high was continuously fed by  
161 nanofiltration permeate thanks to a peristaltic pump (BT100F-1, Lead Fluid) set at 1.1L per day  
162 flow for 24 days. The flow was controlled daily. Daily samples were taken at the outlet of the  
163 soil column as shown in Appendix (see Figure A1).

### 164 **2.3.Parameters studied:**

165 Different parameters were determined during the experiment. The measurement protocol for  
166 each parameter is described below:

167 *2.3.1.Physicochemical parameters:* pH, electrical conductivity (EC), dissolved oxygen ( $O_2$  dis)  
168 and redox potential (eH) were measured during the experiment. Specific cells were built  
169 thanks to a 3D printer to fit with DO sensor and eH electrode. These cells allowed the  
170 measurement of both parameters without contact with the atmosphere. Multimeters were  
171 used for the measurements (WTW, Multi3620 IDS ; Swan, Chematest25 ; Consort,  
172 multiparameter analyser C533). Electrodes and sensors were weekly checked with calibration  
173 solutions (buffer 7 for pH, Hanna 240 or 470mV solution for eH, Hanna 1413  $\mu$ S.cm<sup>-1</sup> for EC).  
174 No shifts were observed during the experiments.

175 2.3.2. *Chemical Oxygen Demand (COD)*: COD kits for low range (0-150 mg(O<sub>2</sub>).L<sup>-1</sup>) were  
176 purchased (LCI500, Hach Lange). 2mL of samples were added in each premade analysis vial.  
177 Vials were heated at 148°C for 2 hours. COD values were obtained by using a  
178 spectrophotometer (DR1900, Hach Lange). COD was determined thanks to the reaction of  
179 Cr<sub>2</sub>O<sub>7</sub><sup>2-</sup> turning into Cr<sup>3+</sup> by reaction with O<sub>2</sub>. Colour turned from orange to green.

180 2.3.3. *Pharmaceuticals and pesticides (CECs)*: A protocol of extraction was performed to  
181 preconcentrate 80 CECs compounds (Brice *et al.*, 2022; Gros *et al.*, 2006; Sellier, 2022). The list  
182 of the studied compounds associated to some of their properties and the limits of  
183 quantification of the method is shown in Appendix (Table A2). Samples of 25mL (WWTP,  
184 permeate, retentate) or 100 mL (WWTP, bottom of the soil column for the coupling  
185 experiment) were collected. These aliquots were spiked with surrogates to ensure the  
186 reliability of the extraction process. After spiking the surrogates, filtration thanks to PVDF  
187 hydrophobic filters with pore diameter of 0.45µm (Merck) were performed. The 25mL and  
188 100mL samples are diluted in 250mL with pure water. Thus, the 250mL were passed through  
189 SPE cartridges (Oasis HLB, 500mg, 6cc). Cartridges were conditioned before using. After the  
190 percolation of the 250mL, CECs were eluted with 3\*3.4mL of methanol. Methanol was totally  
191 evaporated under nitrogen flow at 40°C. Recoveries were performed with a solution  
192 containing internal standard (95% pure water and 5% methanol). 250µL of solution were used  
193 for each samples allowing preconcentration factor of 100 or 400 according to the initial volume  
194 (respectively 25 and 100mL).

195 LC-MS/MS 8040 (Shimadzu) was used to evaluate the concentration of the CECs. Kinetex polar  
196 C18 column was used to separate along time the CECs. Methanol (with 0.1% of formic acid)  
197 and water (with 0.1% of formic acid) were used as solvents with an initial percentage of 5%  
198 and 95% respectively. Gradient was applying as shown in Appendix (Figure A2). Operating

199 conditions of the mass spectrometer was detailed in Appendix (Table A3). Fresh calibrating  
200 solutions, ranging from 0 to 200 $\mu\text{g}\cdot\text{L}^{-1}$ , were used for the quantification. Spectra were treated  
201 with Labsolutions Insights software. Quality controls were performed every 8 samples to  
202 detect potential concentration shifts.

203 *2.3.4. Anions and cations:* Major cations and anions were quantified thanks to ion  
204 chromatography (Metrohm 930 Compact IC Flex, anion column: Metrosep A supp 5 -250/4.0,  
205 cation column/ Metrosep C 6 – A150/4.0). Temperature was set at 35°C for anions and 30°C  
206 for cations. The quantification was performed with a flow rate of 0.7 $\text{mL}\cdot\text{min}^{-1}$  for anions with  
207 a sodium bicarbonate and sodium carbonate eluent (3.2 $\text{mM}$ /1.0 $\text{mM}$ ) respectively. For cations,  
208 the quantification was performed with a flow rate of 0.7 $\text{mL}\cdot\text{min}^{-1}$  with a nitric acid and  
209 dipicolinic acid eluent (1.7 $\text{mM}$ /1.7 $\text{mM}$ ) respectively.

#### 210 **2.4. WWTP effluent:**

211 Effluents were collected in the Baillargues – Saint-Brès WWTP (20,000 inhabitant equivalent).  
212 The WWTP is equipped with a MBR allowing the direct use of the effluent for nanofiltration.  
213 Indeed, silt density index for this WWTP is below 3 (2.5) which allows its direct use for  
214 nanofiltration process (Mendret *et al.*, 2019). Effluents were collected in January 2023.  
215 Effluents were stored at 4°C until use to limit change of composition of the effluent particularly  
216 for the CECs. The physicochemical characteristics of WWTP effluent will be described in the  
217 next section.

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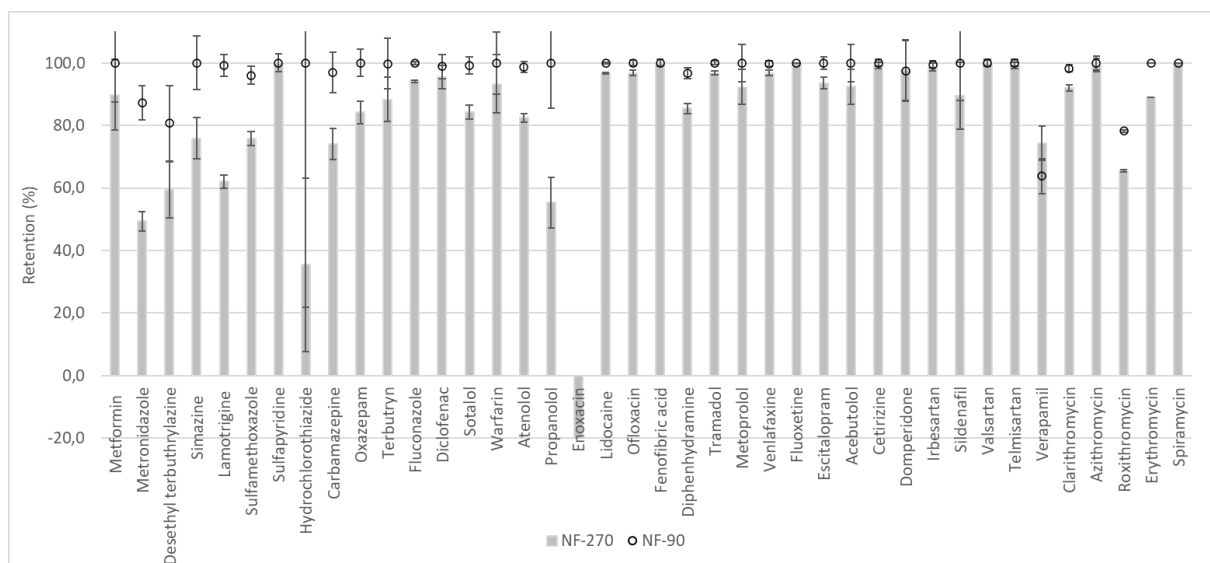
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221 **3.Results and discussion:**

222 **3.1.Nanofiltration performance:**

223 The performances of the two commercial membranes NF-90 and NF-270 were tested using  
224 the MBR effluent. The retention for each CEC, sorted from lower to higher Stokes radius,  
225 detected in the effluent is shown Figure 1.



226

227 **Figure 1.** Comparison of NF-90 and NF-270 performances for the removal of CECs (8 bars, 20°C,  
228 80% conversion).

229 The results agree with previous data found in the literature. NF-90 due to its lower pore radii  
230 is more efficient than NF-270 to remove CECs (Azaïs *et al.*, 2014; Azaïs *et al.*, 2016; Wang *et al.*,  
231 2021; Zhao *et al.*, 2017). The NF-90 is also more efficient for the retention of monovalent ions  
232 and total organic carbon while the retention of total organic carbon and divalent ions are quite  
233 comparable for both membranes according to literature (Azaïs, 2015; Bunani *et al.*, 2013;  
234 Yabalak *et al.*, 2021). As soil infiltration is expected to improve water quality NF-90 is too much  
235 efficient to be used for coupling treatment. NF-270 has shown interesting performances of  
236 retention for all the parameters studied. This membrane was thus chosen for performing  
237 coupling experiments due to its higher permeability and lower cost of operation and

238 maintenance in comparison with NF-90 (Azaïs, 2015; Mendret *et al.*, 2019; Ramdani *et al.*,  
239 2021). The influence of transmembrane pressure or conversion rate on the NF-270 removal  
240 efficiency for CECs was also investigated. Few effects on the retention have been observed as  
241 shown in Appendix (Figure A3). Indeed, these effects remain slight regarding the global  
242 retention of the CECs, modifying only the retention efficiency of some CECs, as already  
243 demonstrated in literature (Giacobbo *et al.*, 2023; Gur-Reznik *et al.*, 2023; Plantard *et al.*,  
244 2018).

### 245 **3.2. Soil infiltration performance:**

246 A preliminary, non-quantitative experiment, using the same soil and the same column (30cm-  
247 high) has been performed with the effluent to determine the time necessary to detect CECs at  
248 the outlet. The experiment confirmed some patterns observed in the literature. Indeed, it has  
249 been generally described in literature that positively charged compounds are not detected  
250 after soil column infiltration or with a high retardation compared with negatively and neutral  
251 charged compounds (Demeau b; Hermes *et al.*, 2019; Schaffer and Licha, 2015; Schaffer *et al.*,  
252 2015). The same patterns have been observed during our experiments. For the negative and  
253 neutral compounds some of them could be observed at the outlet of the column  
254 (epoxycarbamazepine or carbamazepine for instance) while other compounds were totally  
255 removed such as valsartan. Moreover, breakthrough curves could be delayed according to the  
256 characteristics of the compounds. Soil infiltration has been investigating more carefully during  
257 the coupling treatment.

258

259 **3.3.Coupling experiments:**

260 Physicochemical parameters, major ions, COD and CECs have been determined during the 24  
261 days infiltration experiment. The results obtained for these parameters and discussion about  
262 their evolution are presented below.

263 **3.3.1.Physicochemical parameters variations:**

264 These parameters regroup pH, redox potential (eH), electrical conductivity (EC) and dissolved  
265 oxygen ( $O_2$  dis). The mean values found initially in the effluent of WWTP and after each  
266 treatment step are summarized in Table 1.

267 **Table 1.** Physicochemical characteristics of WWTP effluent, permeate (column feeding) and  
268 the outlet of the soil column.

Parameters	WWTP effluent (n=3)	Column feeding (n=6)	After soil column (n=8)
pH	$7.8 \pm 0.1$	$7.5 \pm 0.3$	$7.9 \pm 0.2$
EC ( $\mu\text{S}\cdot\text{cm}^{-1}$ )	$1410 \pm 10$	$920 \pm 30$	$1017 \pm 14$
$O_2$ dis (%)	$100 \pm 5$	$100 \pm 5$	$8 \pm 4$
eH (mV)	$170 \pm 20$	$200 \pm 30$	$146 \pm 14$

269

270 Few pH differences were observed during the experiment likely due to the pH of the  
271 soil (Table 1).

272 Concerning dissolved oxygen, nanofiltration did not affect the percentage of dissolved  
273 oxygen. Water after the soil column was depleted in oxygen as already observed in literature  
274 for saturated columns, inducing anoxic conditions (Farnsworth *et al.*, 2012; Horner *et al.*, 2007;  
275 Pan *et al.*, 2017).

276 Nanofiltration did not seem to induce a huge change in eH as already reported in  
277 literature (Jährić *et al.*, 2018). However, during soil infiltration, eH seemed to decrease

278 gradually until reaching a plateau around 140-150mV. The decrease observed could be due to  
279 several reasons (i) either the way of the infiltration of the feeding solution (Miele *et al.*, 2023;  
280 Rodriguez-Escales *et al.*, 2020) or (ii) the removal of some bacteria by nanofiltration (Oliveira  
281 *et al.*, 2022) which can be responsible of reductive phenomena (Couture *et al.*, 2015).

282 Finally electrical conductivity was reduced thanks to nanofiltration after the first  
283 treatment likely due to the anions and cations retention. However, after soil infiltration a slight  
284 increase was observed for the electrical conductivity. To have a better understanding of the  
285 phenomena involved during nanofiltration and soil infiltration it is interesting to focus on  
286 major ions which are mainly responsible of the electrical conductivity value.

### 287 3.3.2. Major ions variation:

288 Only the ions with concentrations above 1mg.L<sup>-1</sup> will be presented in this part. Measurements  
289 for anions or cations with concentrations below 1mg.L<sup>-1</sup> were stable during the whole  
290 experiment. The concentrations found in the effluent and after each treatment are  
291 summarized in Table 2.

292 **Table 2.** Concentrations of ions in effluent and after each treatment (mg.L<sup>-1</sup>).

Ions	Cl <sup>-</sup>	NO <sub>3</sub> <sup>-</sup>	SO <sub>4</sub> <sup>2-</sup>	Ca <sup>2+</sup>	K <sup>+</sup>	Mg <sup>2+</sup>	Na <sup>+</sup>
Effluent	231	2.6	86	118	18.7	6.2	152
After NF	217	3.2	3.7	40	14.3	3.2	124
After soil	218	3.2	3.9	76	0.97	3.4	118

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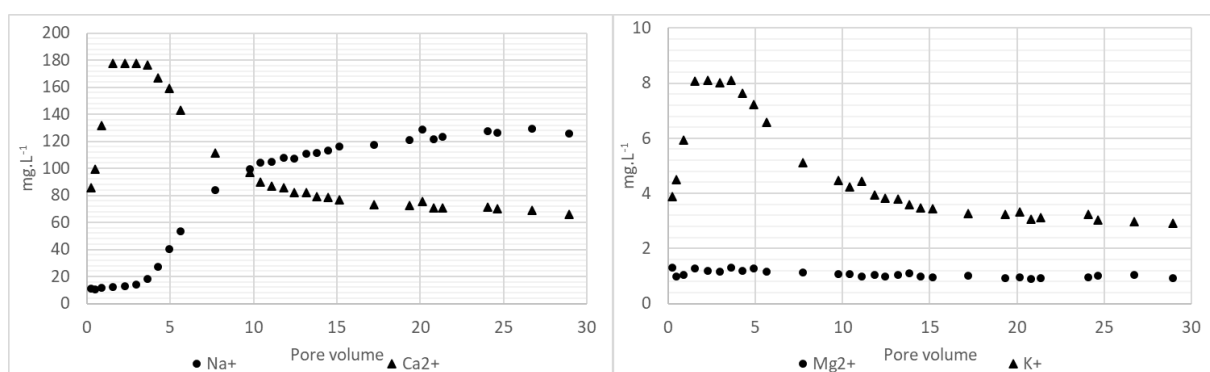
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295 Nanofiltration was particularly efficient for removing divalent ions as already described in  
296 literature (Azais, 2015; Bunani *et al.*, 2013) with removal rates ranging from 48 to 96% for

297 divalent ions while monovalent ion retentions range from 6% to 24%. For nitrate ion no  
298 removal was observed but its concentration (Mendret *et al.*, 2019).

299 Generally, for negative ions no further removal was performed by the soil infiltration. This lack  
300 of removal for anions could be problematic for chloride ions. Indeed, even if no guideline value  
301 is proposed by the WHO (Guidelines for drinking-water quality) some national legislations limit  
302 the value of chloride in water used in purpose of drinking use at  $200\text{mg.L}^{-1}$  and lower  $250\text{mg.L}^{-1}$   
303 <sup>1</sup> in drinking water (Arrêté du 11 janvier 2017; Informe, 2019). This result confirms that anions  
304 could be good tracer for soil infiltration. In some studies, bromide ions are sometimes used as  
305 tracer (Kiecak *et al.*, 2020; Rauch-Williams *et al.*, 2010). However, in our study the level of such  
306 ions is too low, and no additional element was added to maintain natural equilibrium. In this  
307 study, chloride ions can be used as tracer (Guillemoto *et al.*, 2022), to compare breakthrough  
308 speed, as sulfate ions have shown a slight retardation compared with chloride ions.

309 For cations some changes clearly occurred in soil at the start of the infiltration. Excess of  
310 magnesium and calcium ions were observed in solutions during the first pore volumes as  
311 shown Figure 2.



312  
313 **Figure 2.** Evolution of cations concentrations in soil ( $Q = 1.1\text{L.day}^{-1}$ ,  $T \approx 10\text{-}12^\circ\text{C}$ , 24 days,  $\text{pH} \approx$   
314  $7.8\text{-}8$ ).

315 While potassium ions tended to be adsorbed by soil during infiltration, magnesium and  
316 calcium breakthrough curve were fast responsive after the introduction of the effluent. When



317 sodium ions started to breakthrough both magnesium and calcium ions concentrations started  
318 to decrease. Cation exchanges clearly seemed to be involved when nanofiltration permeate  
319 was infiltrated in soil. The soil composition tended to evolve with an adsorption of sodium and  
320 potassium ions and a release of calcium and magnesium ions until it reached an equilibrium.  
321 This kind of exchange has already been reported in previous study using WWTP effluent as  
322 feeding solution (Goren *et al.*, 2011; Guillemoto *et al.*, 2022). The exchange occurred quickly  
323 in the first 10 pore volumes before slowing down while concentrations approaching their  
324 plateau values. The exchanges are dependant of the initial concentrations in soil, the cation  
325 capacity exchange, and the effluent as  $\text{Na}^+$  is sometimes released and  $\text{Ca}^{2+}$  adsorbed as shown  
326 in literature (Bekir *et al.*, 2022; Garcia-Menéndez *et al.*, 2021).

### 327 3.3.3. COD variation:

328 COD is often followed in soil aquifer treatment experiments and is a good indicator of water  
329 quality (Shu *et al.*, 2019). Initial values of COD (close to  $20\text{mg}(\text{O}_2).\text{L}^{-1}$ ) in treated effluent were  
330 already low in comparison to conventional wastewater effluent due to the MBR used at  
331 Baillargues-St-Brès. After the nanofiltration treatment, the concentrations of COD ranged from  
332 3 to  $5\text{mg}(\text{O}_2).\text{L}^{-1}$  showing a good efficiency of nanofiltration membrane to decrease the COD  
333 value. Finally in soil, an increase of COD values was observed, reaching concentrations  
334 between 10 to  $20\text{mg}(\text{O}_2).\text{L}^{-1}$ . Species accounting for COD concentrations were likely present  
335 in soil as an initial value around 50 to  $80\text{mg}(\text{O}_2).\text{L}^{-1}$  has been observed for all the experiment.  
336 At the start of the experiment, water present in the column has been in contact with the soil  
337 for at least 3 days (due to the saturation of the soil column) which could be an explanation for  
338 higher initial values quantified. Usually, soil infiltration allows to decrease the value of COD  
339 (Coutinho *et al.*, 2018; Pan *et al.*, 2017). However, in literature the value of COD is higher than

340 those applied in the infiltration of nanofiltration permeate in soil which could explain the  
341 increase of COD in this study.

#### 342 3.3.4. CECs variation:

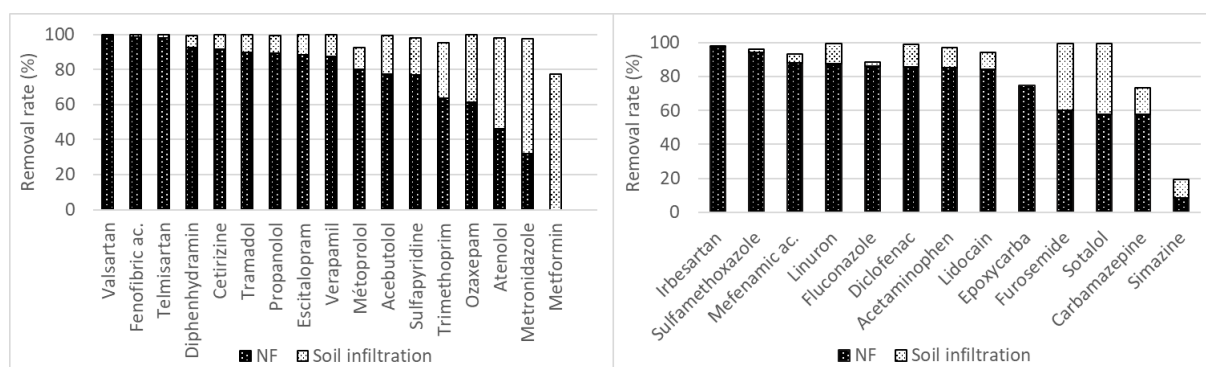
343 Finally, concentrations of 39 CECs have been followed in the treated effluent and during the  
344 different treatment steps to assess the combined removal efficiency of nanofiltration and soil  
345 column infiltration but also the individual removal efficiency for nanofiltration. The removal  
346 efficiency was calculated thanks to the removal rate Equation 1:

$$347 \text{ Removal}(\%) = \frac{C_{\text{effluent}} - C_{\text{permeate}}}{C_{\text{effluent}}} * 100 \text{ (Eq.1)}$$

348 With  $C_{\text{effluent}}$  corresponding to the concentration of CECs in the wastewater treatment plant  
349 effluent and  $C_{\text{permeate}}$  the concentration of the feeding solution after mixing 15L and 17L  
350 permeate solutions corresponding to two days at 80% conversion and 2 days at 90%  
351 conversion with nanofiltration performed at 5 bars and 20°C. The concentrations of CECs in  
352 the feeding solution were determined regularly during the 24 days. No decrease was observed.

353 Nanofiltration shown an overall good removal efficiency with 27 compounds for which  
354 removal reached more than 70% including 5 compounds which were not detected anymore. 7  
355 compounds were partially removed between 30% and 70% by NF and finally 4 compounds;  
356 saccharine, simazine, metformin and thiabendazole; were hardly removed by nanofiltration  
357 process. These compounds have small Stokes radii (see Table A2) which can partly explained  
358 why their removal are lower than other molecules. As shown Figure 1, generally for molecules  
359 with a Stokes radius above 4.84Å (lidocaine) more than 90% removal was observed with few  
360 exceptions. Molecules with small Stokes radii are not removed by steric exclusion. Other  
361 parameters could also be taken into consideration such as the charge or the hydrophobicity of

362 the compounds (Azaïs *et al.*, 2016; Taheran *et al.*, 2016; Wang *et al.*, 2021). Then, soil  
 363 infiltration allowed a good increase of the removal for many compounds. Only three  
 364 compounds had total removal below 70% after this step (saccharine, hydrochlorothiazide, and  
 365 simazine) and 32 compounds out of 39 were removed more than 90% including 22 compounds  
 366 which were not detected at the end of soil column after 24 days. Mainly positive compounds  
 367 were removed, or at least highly retarded during soil infiltration, likely due to the negative net  
 368 charge of the soil as already described in literature (Hermes *et al.*, 2019; Schaffer *et al.*, 2015).  
 369 Moreover, some other compounds such as valsartan or oxazepam were not find at the end of  
 370 the column despite their neutral or negative charge. However, these compounds have already  
 371 shown a good or moderate removal in previous studies (Hermes *et al.*, 2019; Muntau *et al.*,  
 372 2017; Sallwey *et al.*, 2020; Schaffer *et al.*, 2015). Finally, some compounds such as sotalol and  
 373 lamotrigine were still in the process of breaking through soil column and their final  
 374 concentration could be higher than those after 24 days. Figure 3 depicted the influence of each  
 375 treatment on the total removal to determine which compounds are recalcitrant to each  
 376 method of degradation/retention. Table 3 summarizes the initial concentrations and after each  
 377 treatment but also the removal efficiency of the CECs.



378  
 379 **Figure 3.** Removal rate (%) associated to each treatment steps; NF performed at 5 bars, 20°C, 80% and  
 380 90% of conversion; soil infiltration performed for 24 days with concentrations of CECs averaged on the  
 381 last 7 days or when the plateau is reached.

382

383 **Table 3.** Concentrations of the CECs in WWTP effluent and after each treatment. The number under bracket corresponds to the removal in  
 384 comparison with WWTP effluent concentrations. Under parenthesis the main charge of the compounds is indicated (+) for positive, (0) for  
 385 neutral and (-) for negative at pH =7.9.

	Compounds	C eff (ng.L <sup>-1</sup> )	C NF (ng.L <sup>-1</sup> )	C Soil (ng.L <sup>-1</sup> )		Compounds	C eff (ng.L <sup>-1</sup> )	C NF (ng.L <sup>-1</sup> )	C Soil (ng.L <sup>-1</sup> )
Partially removed by the soil column	Acetaminophen (0)	68 ± 60	10 ± 6 (85)	2 ± 2 (97)	Not detected after the soil column	Acebutolol (+)	71 ± 3	16 ± 4 (77)	<5 (>93)
	Carbamazepine (0)	2570 ± 240	1090 ± 200 (58)	686 ± 52 (73)		Atenolol (+)	26 ± 3	14 ± 2 (46)	<1 (>96)
	Diclofenac (-)	1760 ± 110	250 ± 100 (86)	18 ± 33 (99)		Cetirizine (+/0)	256 ± 53	22 ± 2 (91)	<5 (>98)
	Furosemide (-)	375 ± 280	150 ± 40 (60)	1.7 ± 1.5 (100)		Diphenhydramine (+)	13 ± 4	1 ± 1 (92)	<0.1 (>99)
	Lamotrigine (0)	5455 ± 2420	2010 ± 428 (63)	256 ± 90 (95)		Escitalopram (+)	28 ± 6	3,2 ± 1,4 (89)	<0.3 (>99)
	Lidocaine (0/+)	404 ± 37	63 ± 13 (84)	23 ± 5 (94)		Fenofibric ac. (0)	113 ± 50	1,5 ± 2,6 (99)	<0.3 (100)
	Mefenamic ac. (-)	40 ± 31	4.7 ± 2.5 (88)	2.7 ± 1.3 (93)		Linuron (0)	76 ± 7	9.4 ± 2.2 (88)	0.4 ± 0.3 (100)
	Saccharine (-)	63 ± 28	67 ± 74 (-6)	32 ± 38 (49)		Metformin (++)	2,2 ± 0,9	2,2 ± 0,9 (0)	<0.2 (>91)
	Sotalol (+)	1290 ± 70	543 ± 109 (58)	8.8 ± 2.2 (99)		Metoprolol (+)	6,5 ± 0,6	1,3 ± 1,3 (80)	<0,1 (>98)
Not removed by soil column	Epoxy carbamazepine (0)	128 ± 6	32 ± 3 (75)	37 ± 3 (71)		Metronidazole (0)	22 ± 2	15 ± 2 (32)	<2 (>91)
	Fluconazole (0)	176 ± 15	24 ± 3 (86)	20 ± 3 (89)		Oxazepam (0)	1660 ± 250	640 ± 54 (61)	2,2 ± 1,6 (100)
	Hydrochlorothiazide	2940 ± 530	1770 ± 210 (40)	1630 ± 240 (45)		Propranolol (+)	66 ± 17	7 ± 13 (89)	<0.2 (100)
	Irbesartan (-)	11900 ± 4300	292 ± 80 (98)	277 ± 124 (98)		Sulfapyridine (-)	24 ± 3	5,5 ± 1,0 (77)	<1 (>96)
	Simazine (0)	3.6 ± 0.8	3.3 ± 0.3 (8)	2.9 ± 0.6 (19)		Telmisartan (-)	1130 ± 100	20 ± 4 (98)	<0.5 (100)
	Sulfamethoxazole (-)	155 ± 61	8.9 ± 1.5 (94)	6.0 ± 0.5 (96)		Thiabendazole (0)	2,1 ± 1,9	4,2 ± 1,1 (-100)	<0.2 (>90)
Removed by NF	Erythromycin (+)	9.2 ± 3.2	<1 (>90)	/		Tramadol (+)	2140 ± 40	216 ± 50 (90)	<10 (100)
	Gemfibrozil (-)	62 ± 37	<0.5 (100)	/		Trimethoprim (+/0)	10,7 ± 1,6	3,9 ± 0,3 (62)	<1 (>91)
	Ofloxacin (0/-)	245 ± 36	<1 (100)	/		Valsartan (--)	1130 ± 450	13 ± 4 (99)	<0.2 (100)
	Spyramycin (++)	104 ± 24	<0.5 (100)	/		Venlafaxine (+)	690 ± 90	86 ± 7 (88)	<5 (100)
	Verapamil (+)	15 ± 6	<0.3 (>98)	/					

386 NF: 5 bars, 20°C, homogenised solution of permeate at 80% conversion (15L) and 90% conversion (17L).

387 Soil infiltration: Q = 1.1L.day<sup>-1</sup>, T ≈ 10-12°C, 24 days.

388

389 Soil infiltration appears to be a good complementary treatment after nanofiltration performed  
390 with the NF-270 membrane. If the membrane was responsible of 80% (Arola *et al.*, 2017) of  
391 the total removal when the concentrations of all CECs are summed in this study, close to  
392 percentage found in previous study. The soil infiltration allowed to raise this percentage up to  
393 92% showing a very efficient coupling advanced treatment. For trimethoprim, oxazepam  
394 metronidazole, furosemide, atenolol, and sotalol the removal, for both techniques, was similar  
395 and the final removal was close to 100%. For simazine, nanofiltration and soil infiltration have  
396 shown limitations with a removal of only 20% at the coupling treatment. Simazine in soils has  
397 already been described as a persistent compound (Trussell *et al.*, 2018) but its recalcitrant  
398 behaviour with NF-270 membrane has not been reported yet. Even if simazine had 70%  
399 removal rate with NF-270 at 80% of conversion, this removal dropped at 35% when the  
400 conversion rate was increased at 90%. It explains the high concentration of simazine in the  
401 permeate. Metformin was the only compound for which nanofiltration was not efficient at all  
402 with no change of concentration between the effluent of the WWTP and the permeate. In this  
403 case, soil infiltration was very efficient for eliminated the metformin as already observed  
404 elsewhere (Hellauer *et al.*, 2018). Thiabendazole was also not removed by nanofiltration and  
405 even more concentrated in the permeate than in the concentrate. It is the first time to our  
406 knowledge that thiabendazole recalcitrant behaviour is shown for nanofiltration. However,  
407 thiabendazole concentration was low and the molecule was removed by soil.

#### 408 3.3.5. Removal of CECs in coupling experiments in the literature:

409 In literature some articles have already investigated the efficiency of coupling some advanced  
410 treatments on the removal of CECs. For instance, Shafdan site, in Israel, have already  
411 performed SAT coupling with membrane process (Cikurel *et al.*, 2006) or ozonation (Zucker *et*

412 al., 2015; Lakretz *et al.*, 2017). Indeed, coupling of UF and short SAT or short SAT and NF-270  
413 have been performed (Cikurel *et al.*, 2006). In both case the removal obtained for the  
414 compounds are higher than those traditionally observed with conventional SAT applied on site.  
415 In comparison with our experiment only sulfamethoxazole and trimethoprim are initially  
416 observed in the effluent likely due to the MBR treatment applied by the Baillargues-St-Brès  
417 WWTP in comparison with the conventional activated sludge used in Shafdan (Hermes *et al.*,  
418 2019). For both compounds removals are in the same order between our experiment and  
419 Shafdan experiment with almost 100% of removal for trimethoprim and >90% for  
420 sulfamethoxazole. Concentrations ranged from 173 to 657ng.L<sup>-1</sup> for sulfamethoxazole (like our  
421 study) and 62 to 349ng.L<sup>-1</sup> for trimethoprim (above the concentration of this study).

422 Combination of ozonation and short SAT has also been investigated in Shafdan. For diclofenac  
423 and sulfamethoxazole, their removal is almost complete. Removal of metoprolol and  
424 venlafaxine respectively reaches around 90% and 80 to 97% respectively slightly lower than  
425 those obtained with our coupling. Finally, carbamazepine tends to be totally removed by  
426 ozonation, but retardation of carbamazepine can induce high concentration during several  
427 weeks before reaching concentrations below limit of quantification (Lakretz *et al.*, 2017;  
428 Zucker *et al.*, 2015).

429 In general, ozonation has shown very promising results when coupled with other techniques  
430 (Hellauer *et al.*, 2017; Kim *et al.*, 2019; Lakretz *et al.*, 2017; Real *et al.*, 2012; Sharma *et al.*,  
431 2017; Zucker *et al.*, 2015). Dose of ozone used ranged from 0.6mgO<sub>3</sub>/mg(DOC) (Hellauer *et al.*,  
432 2017) to 1-1.2 mgO<sub>3</sub>/mg(DOC) (Kim *et al.*, 2019; Lakretz *et al.*, 2017; Sharma *et al.*, 2017; Zucker  
433 *et al.*, 2015). For carbamazepine, with concentration close to our study from 700 to 1000ng.L<sup>-1</sup>  
434 <sup>1</sup>, the removal is higher than those obtained with nanofiltration as carbamazepine is totally

435 degraded by ozonation. These studies (Hellauer *et al.*, , 2017; Lakretz *et al.*, 2016 ; Zucker *et*  
436 *al.*, 2015) confirm removal of venlafaxine (250-750ng.L<sup>-1</sup>), is good with ozonation (at least 80-  
437 90%) with similar concentrations than our study. The removal is sometimes not total, but its  
438 concentration is often under limit of quantification (20ng.L<sup>-1</sup>). For diclofenac (300-1300ng.L<sup>-1</sup>),  
439 the removal is complete like in this study (99%). Metoprolol (300-1000ng.L<sup>-1</sup>), totally removed  
440 in our study but with a much lower initial concentration (6ng.L<sup>-1</sup>), can show different  
441 behaviours as it is sometimes almost removed (>98%) (Hellauer *et al.*, , 2017; Zucker *et al.*,  
442 2015), sometimes poorly removed when oxidation treatment is used before NF (O<sub>3</sub> + NF and  
443 O<sub>3</sub>/H<sub>2</sub>O<sub>2</sub> + NF) and totally removed with nanofiltration followed by ozonation<sup>79</sup>. Finally,  
444 hydrochlorothiazide which have shown a recalcitrant behaviour in our experiment with 45%  
445 removal, with initial concentration around 3µg.L<sup>-1</sup> in this study, is totally removed when  
446 applying UV treatment and nanofiltration but also poorly removed with other combination  
447 such as NF and ozonation or chlorination with respectively 55% and 65% of removal but with  
448 concentration close to 0.3mg.L<sup>-1</sup> (1µM) (Real *et al.*, 2012) . H<sub>2</sub>O<sub>2</sub> is sometimes used to boost  
449 performances of the oxidation such as ozonation, Fenton-like reaction or UV irradiation in  
450 addition of coupling with other advanced treatment (Kim *et al.*, 2019; Usman *et al.*, 2018;  
451 Wünsch *et al.*, 2019). However, the increase of performance is generally slight.

452 Finally, some soil aquifer treatments, corresponding to the infiltration of treated wastewater  
453 through the surface and vadose zone followed by a slow transport in the aquifer, are somehow  
454 enhanced with reactive layer or a better optimisation of the process (Hernandez Garcia *et al.*,  
455 2014; Lakretz *et al.*, 2017; Liu *et al.*, 2020 Schaffer *et al.*, 2015; Trussell *et al.*, 2018; Valhondo  
456 *et al.*, 2020). Even if these experiments are not exactly “coupling process” because the  
457 mechanisms of removal are close, the addition of reactive layer or a constructed wetland can

458 improve the performance of removal for some compounds such as benzotriazole,  
459 epoxycarbamazepine, gemfibrozil or sulfamethoxazole.

#### 460 **4.Conclusions:**

461 The efficiency of a novel combination of advanced treatments applied on MBR effluent of  
462 WWTP has been investigated through different parameters such as physicochemical  
463 parameters or concentrations of CECs, particularly problematic in the last decades. Few  
464 changes have been observed concerning physicochemical parameters with only a slight  
465 decrease of electrical conductivity due to nanofiltration process. The introduction in the soil  
466 column of the permeate, collected thanks to the nanofiltration of the MBR effluent, induces a  
467 cationic exchange which can potentially modified the adsorption properties of the soil  
468 regarding CECs. Nanofiltration with NF-270 membrane provides good removal efficiency with  
469 80% of retention of the summed concentration of CECs. Molecules with diameter above pore  
470 radii of the membrane are, with few exceptions, totally removed by steric exclusion while  
471 molecules with lower Stokes radii than the membrane pore radii have removal percentage  
472 depending on other parameters (charge, log  $K_{oc}$ ). Coupling nanofiltration and soil infiltration  
473 has shown a very good efficiency with a reduction of 92% of the overall CECs concentration  
474 allowing an important increase of the removal in comparison with nanofiltration used alone.  
475 This reduction is comparable or even better than some coupling treatments already  
476 investigated in literature such as oxidation process. Furthermore, 22 CECs out of the 39 found  
477 in the effluent are not detected anymore after the coupling and only 4 compounds have  
478 concentration above  $100\text{ng.L}^{-1}$  (*versus* 20 initially) at the end of the treatment. This innovating  
479 study validates the use of nanofiltration and soil infiltration as a promising advanced treatment  
480 coupling according to the results obtained. Improvements of the coupling can still be achieved



481 by adding reactive layer to soil infiltration or optimising the parameters of nanofiltration for  
482 instance. Investigations with longer column feeding should be performed to validate the  
483 preliminary results obtained in this study and observed if soil acclimatisation to permeate  
484 infiltration can further reduce the concentrations of some recalcitrant species due to microbial  
485 population changes.

486

487 **Appendix:** Scheme of the experimental setup, properties of CECs and soil, conditions of LC-  
488 MS/MS quantification and additional experimental results (Word).

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498

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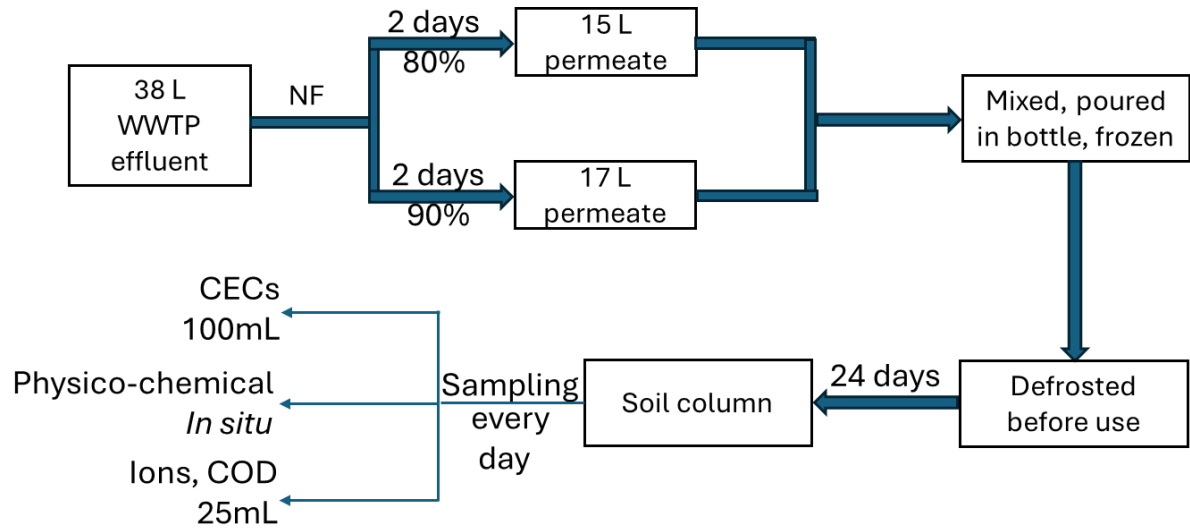
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### Appendix

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894 **Figure A1.** Scheme of the experimental set-up.

895

896 **Table A1.** Soil composition and column characteristics.

897

Soil composition	Clay silty loam (clay 36%; loam 37%; sand 27%)
Soil pH	8.2
TOC	1.2%
Cation exchange capacity Metson	213 me.kg <sup>-1</sup>
Water retention	60%
Pore volume	860mL
Maximal infiltration flowrate	280mL.h <sup>-1</sup>
Working flowrate	46mL.h <sup>-1</sup>

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900

901 **Table A2.** Contaminants of emerging concern sorted by molecular weight and their  
902 characteristics.

	Log P	Log D 7.4	Log D 8	Charge (7,4)	Charge (8)	M molaire (g.mol <sup>-1</sup> )	Stokes r (A°)	Min radius (A°)	Max radius (A°)	Dipole moment (D)	LOQ (ng.L <sup>-1</sup> )
Metformin	- 0,92	-5,62	-5,37	2	1,99	129,167	2,90	3,42	4,92	2,97	0.2
Acetaminophen	0,91	0,9	0,89	-0,01	-0,03	151,17	3,22	3,49	5,67	2,77	0.3
Metronidazole	- 0,46	-0,46	-0,46	0	0	171,15	3,17	4,68	4,75	3,88	2
Saccharine	0,45	-0,49	-0,49	-1	-1	183,2	3,18	3,88	4,62	4,36	10
Thiabendazole	2,33	2,33	2,33	0	-0,01	201,25	3,58	4,01	6,16	2,7	0.2
Simazine	1,78	1,78	1,78	0	0	201,66	3,72	4,9	6,28	5,85	0.1
Carbamazepine	2,95	2,95	2,95	0	0	236,27	4,20	4,48	5,76	3,72	1
Mefenamic acid	4,4	2,29	2,02	-0,98	-1	241,29	4,38	4,92	6,43	1,93	1
Linuron	2,68	2,68	2,68	0	0	249,1	4,18	4,08	6,49	4,99	0.2
Sulfapyridine	1,01	0,24	0,12	-0,94	-0,98	249,29	4,02	4,54	5,89	9,93	1
Gemfibrozil	4,39	1,51	1,14	-1	-1	250,33	4,88	4,99	7,43	2,08	0.5
Epoxy carbamazepine	2,58	2,58	2,58	0	0	252,27					0.1
Sulfamethoxazole	0,79	-0,07	-0,13	-0,97	-0,99	253,28	4,01	5,4	5,88	8,28	0.5
Diphenhydramine	3,65	2,17	2,63	0,97	0,88	255,361	4,99	5,73	6,79	0,99	0.1
Lamotrigine	1,93	1,91	1,92	0,23	0,07	256,09	3,87	3,99	6,1	4,11	30
Propranolol	2,58	0,73	1,3	0,99	0,95	259,35	4,84	4,66	7,41	2,16	0.2
Tramadol	2,45	0,48	1,05	0,99	0,96	263,381	5,02	4,75	7,3	2,88	10

Atenolol	0,43	-1,43	-0,86	0,99	0,95	266,34	4,84	4,19	9,12	6,13	1
Sotalol	-0,84	-1,98	-1,45	0,95	0,82	272,363	4,63	4,21	7,94	5,98	1
Venlafaxine	2,74	1,37	1,91	0,96	0,85	277,408	5,14	5,37	6,79	1,15	5
Oxazepam	2,92	2,92	2,92	0	-0,02	286,72	4,40	5,41	5,76	3,81	0.4
Trimethoprim	1,28	1,1	1,23	0,85	0,59	290,32	4,64	4,97	6,95	4,24	1
Diclofenac	4,26	1,1	0,86	-1	-1	296,15	4,46	4,62	6,34	2,65	0.4
Hydrochlorothiazide	-0,58	-0,58	-0,61	-0,03	-0,1	297,74	4,05	4,13	5,67	8,83	50
Fluconazole	0,56	0,56	0,56	0	0	306,27	4,44	5,1	5,88	3,99	1
Fenofibric acid	5,28	5,28	5,28	0	0	318,75	4,97	4,33	9,72	7,5	0.3
Escitalopram	3,76	1,41	1,98	1	0,98	324,399	5,27	5,76	8,04	3,68	0.3
Furosemide	1,75	-1,63	-1,74	-1	-1,01	330,74	4,45	4,77	7,31	5,96	0.2
Acebutolol	1,53	-0,31	0,26	0,99	0,95	336,432	5,59	4,77	10,69	7,11	5
Ofloxacin	-1,06	-1,08	-1,17	-0,08	-0,29	361,37	4,93	5,27	8,2	9,9	1
Cetirizine	0,86	0,78	0,61	0,78	0,61	388,892	5,74	6,19	9,18	3,81	5
Irbesartan	5,39	4,23	4,04	-0,97	-0,99	428,53	5,89	5,86	9,33	1,87	10
Valsartan	5,27	1,08	0,5	-1,97	-1,99	435,52	6,21	5,7	8,66	10,74	0.2
Verapamil	5,04	3,22	3,79	0,99	0,94	454,611	6,92	6,63	8,28	10,05	0.3
Telmisartan	6,13	4,86	4,52	-0,97	-0,99	514,62	6,78	7,85	8,9	7,62	0.5

Metoprolol	1,76	-0,1	0,47	0,99	0,95	534,738	5,10	4,39	10,07	5,53	0.1
Erythromycin	2,6	0,99	1,55	0,98	0,91	733,93	9,06	6,83	9,32	3,31	1
Spiramycin	2,5	-0,5	0,59	1,9	1,68	843,06	9,22	7,5	12,42	10,22	0.5

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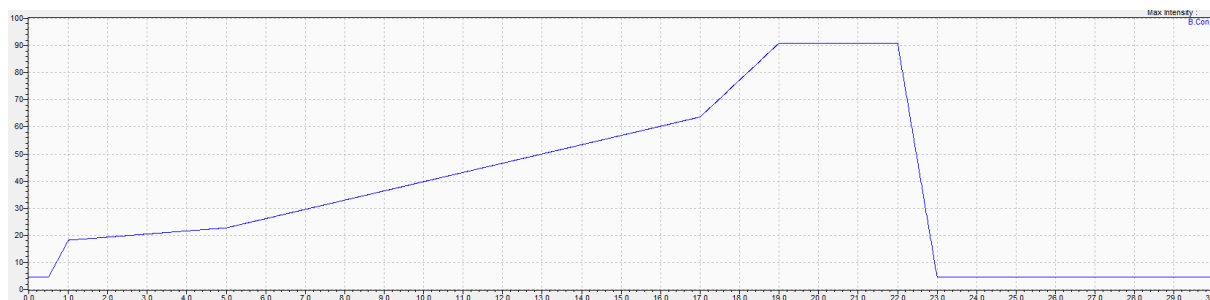
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913 **Figure A2.** Gradient of eluting solution used during liquid chromatography in % of methanol  
 914 + 0.1% formic acid = f(t) in minute



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916 **Table A3.** Operating conditions of mass spectrophotometer

Nebulizing gas flow	2L.min <sup>-1</sup>
DL Temperature	150°C
Heat block temperature	400°C
Drying gas flow	5L.min <sup>-1</sup>

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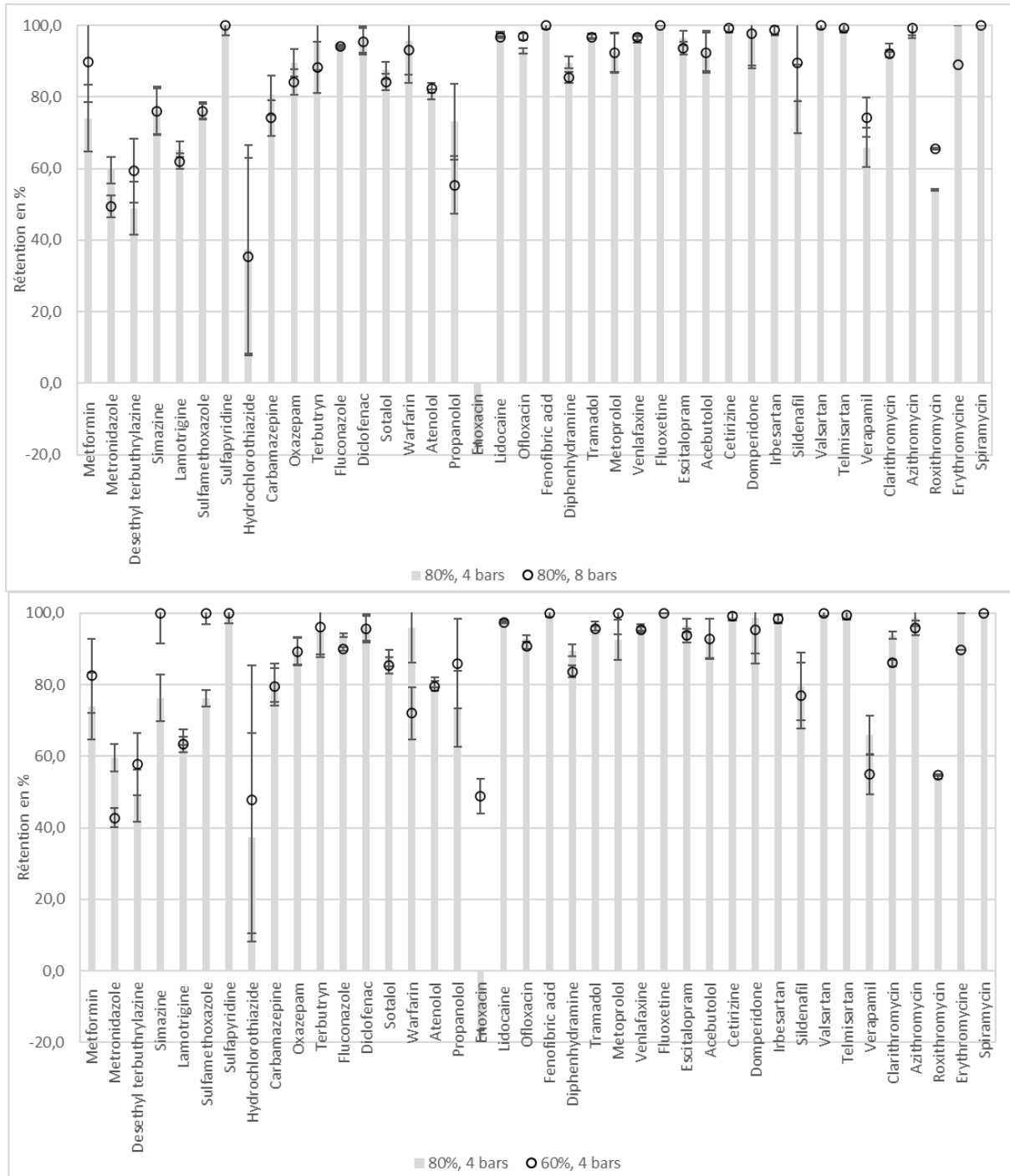
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**Figure A3.** Influence of the retention of NF-270 of CECs for different transmembrane pressure (top) and different conversion rate (bottom)

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