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# **Remediation of soils contaminated by hydrophobic organic compounds: How to recover extracting agents from soil washing solutions?**

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## 1 **Abbreviations**

2 AOP: advanced oxidation process; BDD: boron-doped diamond; CD: cyclodextrin; CMC:  
3 critical micelle concentration; EA: extracting agent; EF: electro-Fenton; HPCD:  
4 hydroxypropyl-  $\beta$  -cyclodextrin; HOC: hydrophobic organic compound;  $K_{CD}$ : complex stability  
5 (or equilibrium) constant between organic compounds and cyclodextrin;  $K_m$ : micellar phase /  
6 aqueous phase partition coefficient; MEUF: micellar-enhanced ultrafiltration process; NOM:  
7 natural organic matter; PAH: polycyclic aromatic hydrocarbon; PHE: phenanthrene; SF soil  
8 flushing; SW: soil washing; TX100: Triton X-100; TW80: Tween<sup>®</sup> 80; VOC: volatile organic  
9 compound.

10

## 11 **Abstract**

12 A lot of soil (particularly, former industrial and military sites) has been contaminated by various  
13 highly toxic contaminants such as petroleum hydrocarbons, polycyclic aromatic hydrocarbons  
14 (PAHs), polychlorobiphenyls (PCBs) or chlorinated solvents. Soil remediation is now required  
15 for their promotion into new industrial or real estate activities. Therefore, the soil washing (SW)  
16 process enhanced by the use of extracting agents (EAs) such as surfactants or cyclodextrins  
17 (CDs) has been developed for the removal of hydrophobic organic compounds (HOCs) from  
18 contaminated soils. The use of extracting agents allows improving the transfer of HOCs from  
19 the soil-sorbed fraction to the washing solution. However, using large amount of extracting  
20 agents is also a critical drawback for cost-effectiveness of the SW process. The aim of this  
21 review is to examine how extracting agents might be recovered from SW solutions for reuse.  
22 Various separation processes are able to recover large amounts of extracting agents according  
23 to the physicochemical characteristics of target pollutants and extracting agents. However, an  
24 additional treatment step is required for the degradation of recovered pollutants. SW solutions

25 may also undergo degradation processes such as advanced oxidation processes (AOPs) with *in*  
26 *situ* production of oxidants. Partial recovery of extracting agents can be achieved according to  
27 operating conditions and reaction kinetics between organic compounds and oxidant species.  
28 The suitability of each process is discussed according to the various physicochemical  
29 characteristics of SW solutions. A particular attention is paid to the anodic oxidation process,  
30 which allows either a selective degradation of the target pollutants or a complete removal of  
31 the organic load depending on the operating conditions.

32

### 33 **Keywords**

34 Soil washing; Reuse; Extracting agent; Separation; Degradation; Hydrophobic organic  
35 pollutant

36

### 37 **Outline**

38 I – Introduction: why should we try to recover extracting agents?

39 II – Separation processes

40 II.1 – Air stripping and related technologies

41 II.2 – Liquid - liquid extraction

42 II.3 – Membrane processes

43 II.4 – Adsorption

44 III – Degradation processes

45 III.1 – Biological processes

46 III.2 – Chemical, photochemical and electrochemical advanced oxidation processes

47 III.2.1 – Removal of target pollutants: generation of oxidant species and reaction  
48 mechanisms

49 III.2.2 – Effectiveness for the recovery of extracting agents

50	III.3 - Soil washing solution reuse or total removal of the organic load? A focus on the
51	possibilities offered by the anodic oxidation process
52	IV – Discussion
53	V – Conclusions and Recommendations
54	Acknowledgements
55	References
56	

## 57 **I – Introduction: why should we try to recover extracting agents?**

58           The development of industrial activities led to the release into the environment of  
59 various hydrophobic organic compounds (HOCs) of environmental concern. Particularly,  
60 polycyclic aromatic hydrocarbons (PAHs), petroleum hydrocarbons, chlorinated solvents and  
61 polychlorobiphenyls (PCBs) are among the most widespread contaminants [1,2]. These  
62 pollutants have a severe toxic, carcinogenic and mutagenic potential to higher organisms,  
63 including humans [3]. Therefore, implementation of human activities at the vicinity of former  
64 industrial sites requires the development of sustainable remediation techniques. This is a major  
65 issue for both public authorities (healthcare risk management) and environmental engineering  
66 companies (development of environmental-friendly and cost-efficient soil remediation  
67 technologies). Conventional remediation techniques all present some major drawbacks such as  
68 low effectiveness (bioremediation in case of aged contaminated soils), high costs (thermal  
69 treatment or excavation) or selectivity towards target pollutants (e.g. volatile organic  
70 compounds (VOCs) for venting) [1,4].

71           Soil is a porous and solid complex matrix made of a mixture of mineral particles and  
72 organic matter [5,6]. The presence of various carbonaceous phases in soils can also be  
73 associated to the specific history of each contaminated site and can lead to strong HOCs  
74 sequestration [7–10]. Using extracting agents (EAs) appeared to be a promising technique in  
75 order to improve the transfer of HOCs from the soil-sorbed fraction to the washing solution  
76 [11,12]. The pump and treat treatment technique enhanced by the use of extracting agents is  
77 named as soil flushing (SF). However, this *in-situ* technique is more difficult to implement and  
78 to control because of soil heterogeneities for several critical parameter such as permeability,  
79 porosity, nature and concentration of pollutants [13]. Therefore, a preliminary excavation step  
80 is often required. A step of granulometric separation is then carried out in order to perform the  
81 chemically enhanced soil washing (SW) process on the most contaminated fraction. Compared

82 to SF, homogenization of the soil allows for optimizing the contact between extracting agents  
83 and soil pollutants. Thus, treatment efficacy is more easily monitored and the contact time is  
84 reduced during SW [1,11].

85 A large range of extracting agents has been tested in the scientific literature. The use of  
86 synthetic surfactants above their critical micelle concentration (CMC) constitutes the most  
87 widespread technique [12,14]. The accumulation of HOCs in the SW solution originates from  
88 the reduction of the interfacial tension between non-aqueous phase liquid (NAPL) and aqueous  
89 phase as well as the increase of solubility due to partitioning of HOCs inside the hydrophobic  
90 space formed by micelles [15,16]. Synthetic surfactants are divided into four main categories,  
91 including anionic (e.g., sodium dodecylsulfate [17,18]), cationic (e.g., quaternary ammonium  
92 derivatives [18]), amphoteric (e.g., cocamidopropyl hydroxysulfate [19]) and non-ionic  
93 surfactants (e.g., Brij<sup>®</sup> 35 [20,21], Tween<sup>®</sup> 80 (TW80) [22–24], Triton<sup>®</sup> X 100 (TX100) [25]).  
94 Surfactant solubilization of organic contaminants can be assessed by a micellar phase / aqueous  
95 phase partition coefficient  $K_m$  (Eq. 1) [15]. The extraction capacity of extracting agents is not  
96 the only parameter to consider for selecting a suitable compound. In fact, some surfactants can  
97 have adverse effect on soil according to their chemical structure [26,27].

$$K_m = \frac{X_m}{X_a} \quad (\text{Eq. 1})$$

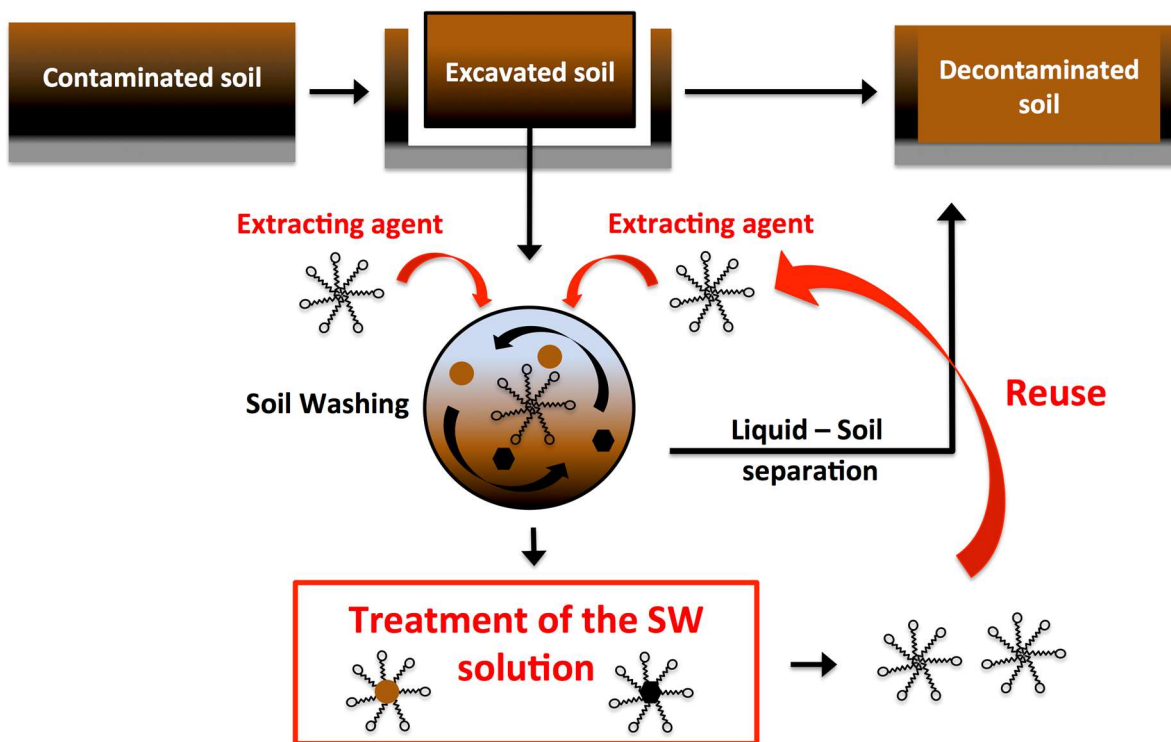
98 where  $X_m$  and  $X_a$  are the mole fraction of target pollutants in the micellar pseudo-phase and  
99 aqueous phase, respectively.

100 There is therefore an important challenge for chemical engineers to develop efficient,  
101 low-cost and eco-friendly surfactants. By comparison to synthetic surfactants, biosurfactants  
102 (from microbial origin) present several advantages such as high biodegradability, lower  
103 toxicity, ecological compatibility and the possibility to be produced *in situ* [28,29]. However,

104 using biosurfactants at industrial scale is still impaired by high production cost and difficulties  
105 to stimulate their *in situ* production at large scale [30,31]. Cyclodextrins (CDs) derivatives (with  
106 higher solubility than native CDs) have also been tested as extracting agents [11,32]. They  
107 allow increasing aqueous solubility of HOCs by forming inclusion complex according to their  
108 respective complex stability (or equilibrium) constant ( $K_{CD}$ ) with organic compounds [11].  
109 However, they are considered to be about ten times less effective than synthetic surfactants in  
110 terms of pollutant removal from soil [11]. Other promising alternatives currently under  
111 development include the use of gemini surfactant (made of two hydrophilic head groups and  
112 two hydrophobic tails) [14,33] and surfactant foams [13,34].

113         Efficient removal of HOCs from soil by EA-enhanced SW depends on the nature of  
114 target pollutants and soil characteristics [11,12]. This process results in the production of highly  
115 concentrated effluents, containing target HOCs at toxic concentrations, natural organic matter  
116 (NOM) from the soil as well as large amount of extracting agents [1]. Therefore, a suitable  
117 strategy for disposal of the SW solution must be implemented [1]. The ecological footprint and  
118 cost-efficiency of the whole process (*i.e.* soil excavation, SW, treatment of the SW solution,  
119 Figure 1) depend strongly on both nature and amount of the surfactant consumed. As eco-  
120 friendly surfactants are usually more expensive, the use of large amounts constitute a major  
121 drawback for further full-scale application of this treatment strategy [1].





122

123 Figure 1– The different steps involved in the treatment of contaminated soils by integrated soil washing  
 124 processes.

125

126 Optimizing the SW process is the most direct way to reduce the consumption of  
 127 extracting agents. Decontamination of polluted soils by SW has already been widely reviewed  
 128 in the literature by using either surfactants [14–16,24] or CDs [11] as extracting agents. The  
 129 most important parameters to consider are: (i) the extraction effectiveness of the chosen EA,  
 130 (ii) its cost, (iii) its soil sorption capacity (which can lead to important loss of extracting agent  
 131 and adverse effects on soil quality) and (iv) the operating parameters, including concentration  
 132 of EA, soil/liquid ratio, contact time, mixing conditions and number of successive washing  
 133 steps [11]. Usually, optimal parameters are determined through preliminary tests. It is worth  
 134 noting that the characteristics of the contaminated soil and pollutants have to be carefully  
 135 investigated in order to assess the suitability of the SW process. According to the contamination  
 136 history, soil pollution ageing and pollutant sequestration can strongly affect the availability of  
 137 target pollutants for extracting agents [8,10,35–38]. Organic matter and clay content might also

138 affect process effectiveness because of competitive solubilization of NOM, mobilization of  
139 hardly settling particles or high adsorption of extracting agents on soil [15]. Finally, the  
140 horizontal and vertical location of the contaminated soil has also to be carefully studied before  
141 soil excavation. Excavation and treatment of non-polluted soil strongly increase the cost of the  
142 process.

143         Once the SW has been optimized, it remains another way to further reduce the  
144 consumption of extracting agents. Several research articles and one previous review paper  
145 focused on the removal of the organic load from SW solutions [1]. However, a large amount of  
146 extracting agents can be recovered by implementing a suitable treatment of the SW solution.  
147 The objective is to improve the cost-efficiency and ecological footprint of soil remediation by  
148 reusing the SW solution. However, it represents an additional treatment step requiring extra-  
149 costs [39,40]. For this reason, this article aims at critically reviewing how to recover extracting  
150 agents during the treatment of SW solutions, including separation and degradation processes.  
151 Different processes are presented and their effectiveness for selective separation or degradation  
152 of target pollutants is discussed according to the specific characteristics of SW solutions.

153

## 154 **II – Separation processes**

### 155 **II.1 – Air stripping and related technologies**

156         The SW process is usually not applied for the treatment of soil contaminated by VOCs  
157 because of the important risk of accidental release. However, surfactant-enhanced SW can be  
158 applied to soils contaminated with pollutants such as trichloroethylene, tetrachlorethylene or  
159 perchloroethylene. In such case, air stripping and related technologies are relevant for the  
160 removal of such compounds from SW solutions [41–44]. Air stripping is based on the formation  
161 of a large air-water interfacial area in order to improve the mass transfer of semi-volatile

162 compounds from the aqueous phase to the gas phase. Volatile or semi-volatile pollutants are  
163 readily partitioned into the gas phase in contrast to non-volatile extracting agents. However,  
164 gas injection in the presence of extracting agents can lead to significant foam formation [45]  
165 which is considered as one of the main drawback of this process [46]. The use of hollow fiber  
166 membranes instead of usual packed towers and tray air strippers can reduce these foaming  
167 issues [47,48]. A lower formation of foam has also been observed with CDs compared to  
168 synthetic surfactants. Air stripping was successfully performed at field-scale for recycling  
169 hydroxypropyl-  $\beta$  -cyclodextrin (HPCD) for several successive SF steps [32,49]. Then,  
170 contaminated air was treated through a granular activated carbon reactor.

171 Solubilization of target pollutants inside surfactant micelles or inclusion complex with  
172 CDs has an important impact on the efficiency of the process. While the higher interfacial  
173 resistance is often considered to only slightly decrease the global mass transfer [45,50],  
174 extracting agents greatly reduce the molar fraction of free organic compounds in the aqueous  
175 phase. Extracting agents act as a competing pseudo-phase for partitioning of the organic  
176 contaminant in the gas phase [51,52]. This adverse effect increases with the hydrophobicity of  
177 target pollutants, solubilization capacity of extracting agents (high  $K_m/K_{CD}$ ) [45] and  
178 concentration of extracting agent [44]. For example, tetrachlorethylene removal rate was 83, 61  
179 and 37% when performing air stripping with hexadecylsulfonate as extracting agent at 0, 10  
180 and 55 mM, respectively [45]. Thus, Lipe et al. (1996) have developed a surfactant air stripping  
181 model (SASM) for assessing the efficiency of air stripping systems by taking into account the  
182 effect of surfactants. It is based on the conventional non-equilibrium mass transfer model and  
183 has been successfully validated by experiments at both bench and field scale [45,48].

184 Vacuum processes have been investigated in order to improve the effectiveness of air  
185 stripping [53]. Vacuum is used as the driving force for the transfer of target compounds from  
186 the liquid to the gas phase. The surfactant stream can also be heated in order to increase the

187 vapor pressure of contaminants. The two main advantages of this process are the higher  
188 separation effectiveness for contaminants with a lower volatility and the reduction of foaming  
189 issues [45].

190 Pervaporation has also been successfully applied to SW solutions [54] containing  
191 tetrachlorethylene [55], trichloroethylene [56], 1,1,1-trichloroethane [54,57] and toluene [57].  
192 It is based on the vaporization of the SW solution through a selective non-porous polymeric  
193 membrane. Field demonstration and pilot-scale experiments demonstrated the high  
194 effectiveness of the process for the removal of these target compounds without affecting the  
195 performance of the recovered solution [55,57]. Different configurations can be used such as  
196 spiral wound modules [57] or hollow fiber membranes [56]. Similarly to previous processes,  
197 the use of high concentrations of extracting agents can decrease the performances in terms of  
198 selectivity and flux [54,55].

199

## 200 **II.2 – Liquid - liquid extraction**

201 An organic liquid can be used as the partitioning phase for the extraction of target HOCs  
202 from SW solutions [44]. Liquid-liquid extraction is based on the mass transfer of target  
203 pollutants from the SW solution to the organic solvent. This process has been successfully  
204 applied at both bench and field scale for the removal of semi- and non-volatile organic  
205 compounds having high Henry's constants [58,59]. Using surfactants as EA, it has been  
206 reported a similar extraction capacity for the treated SW solution compared to the initial  
207 solution [58,59]. However, the process can be further complicated by emulsification of the  
208 solvent by the surfactant solution and solubilization of solvent molecules into the micellar  
209 pseudo-phase. Such phenomenon can be reduced by properly selecting the solvent, particularly  
210 by using highly hydrophobic solvents [58–60]. Hollow fiber membranes filled with the  
211 extracting solvent have also been used in order to eliminate emulsification and reduce the

212 amount of solvent consumed [61]. Petitgirard et al. [62] developed different methods in order  
213 to reduce the amount of solvent used. It was based either on micro-emulsion or impregnation  
214 of an organic membrane with oil. Lower amount of solvent allows decreasing the loss of  
215 surfactant and facilitating the disposal of the contaminated solvent [44].

216 Hasegawa et al. [58] developed a model for liquid-liquid extraction in order to predict  
217 the efficiency of this process for surfactant-containing solutions. The model depends on the  
218 solvent / water partition coefficient ( $K_d$ ), micellar phase / aqueous phase partition coefficient  
219 ( $K_m$ ), solvent-water ratio and hydrodynamic conditions (column height, retention time, water  
220 velocity). Particularly, the partition of target pollutants in micelles ( $K_m$ ) is a crucial parameter.  
221 The increase of the surfactant concentration significantly decreases the efficiency of the process  
222 due to the micellar pseudo-phase acting as a competing partitioning phase [58]. Moreover, it  
223 has been observed that the higher the hydrophobicity of target pollutants (high  $K_m$  and  $K_{CD}$ ),  
224 the higher the adverse effects of extracting agents on removal efficiency is [58].

225 This process has also been applied to solutions containing CDs as extracting agents. For  
226 example, liquid-liquid extraction with rapeseed oil was used for regeneration of a SF solution  
227 containing methyl-  $\beta$  -cyclodextrin [62]. This lab-scale experiment allowed the regeneration of  
228 the methyl-  $\beta$  -cyclodextrin solution for continuous SF. After two days of SF, almost complete  
229 decontamination of the spiked sandy soil was achieved (96-98% of PAH removal), pollutants  
230 were concentrated in the organic phase and the loss of methyl-  $\beta$  -cyclodextrin was negligible.

231 It is worth noting that liquid-liquid extraction leads to the production of highly  
232 contaminated solvents, for which the disposal is a real challenge. Moreover, it also involves  
233 high risks of handling (leaking storage tanks, exposure of workers via skin contact or inhalation).  
234 Distillation systems have been applied for solvent/contaminant separation and reuse of the  
235 solvent for further contaminant/EA separation steps [43], but such process represents an  
236 important cost.

237

### 238 **II.3 – Membrane processes**

239 The use of membrane technologies has also been investigated for the treatment of  
240 SW/SF solutions. Most of the studies did not report selective surfactant-contaminant separation.  
241 However, during SF, are often diluted in recovery wells by groundwater. Therefore, membrane  
242 processes can also be used for re-concentration of the SF solution (e.g. after air stripping) before  
243 reinjection into the contaminated zone [48]. For example, by using ultrafiltration membranes,  
244 it was observed 93-99% of surfactant-contaminant retention from a SW solution containing  
245 naphthalene and trichloroethylene as contaminants and linear alkyl diphenyloxide disulfonate  
246 as surfactant [45]. The formation of micelles improves the retention of organic compounds [63].  
247 Such micellar-enhanced ultrafiltration process (MEUF) is based on the formation of colloid-  
248 sized surfactant micelles retained by the ultrafiltration membrane. Only one stud reported a  
249 selective removal of target pollutants by ultrafiltration: 67% of the surfactant was recovered in  
250 the permeate, while more than 90% of polychlorobiphenyls and 83% of oils were retained [64].  
251 This result might be explained by the formation of oil droplets readily rejected by the membrane,  
252 thus leading to a selective separation of surfactant monomers and pollutants.

253

#### 254 **II.4 – Adsorption**

255 Adsorption is a widely used technology and has recently received great attention for the  
256 treatment of SW solutions because of promising results obtained for the recovery of extracting  
257 agents, especially with activated carbon [1,65] (Table 1). Several studies have observed much  
258 higher removal rates of target pollutants than extracting agents because of the higher partition  
259 coefficient, higher hydrophobicity and lower concentration of target pollutants [66,67].  
260 Recently, the use of organo-bentonite and organo-layered double hydroxide has also been  
261 investigated [68,69]. The objective of such material is to provide a strong affinity between  
262 target pollutants and the organic phase within bentonite or layered double hydroxide. Sorbent  
263 materials, pollutants and extracting agents already studied are reported in Table 1.

264 The effectiveness of this process for the recovery of extracting agents can be assessed  
 265 in batch experiments by using the selectivity ratio ( $S$ ) (Eq. 4) [67]. Values reported in the  
 266 literature for synthetic SW solutions usually range between 5 and 100, according to operating  
 267 conditions (concentration/nature of EAs, target pollutant, sorbent material, dose, etc.). As  
 268 regards to the activated carbon dose in batch experiments, low amount (around 2 g L<sup>-1</sup>) was  
 269 reported to be sufficient to achieve 60% removal of 100 mg L<sup>-1</sup> of PHE with a selectivity ratio  
 270 of 10 (initial concentration of TX100 was 5 g L<sup>-1</sup>) [67]. Much higher effectiveness was also  
 271 reached with lower PHE concentration [67]. However, selectivity ratio measured in synthetic  
 272 solutions cannot be used for prediction of the efficiency of the process with real SW solutions,  
 273 in which much more competition effects occur.

$$S = \frac{C_{SM,j}}{C_{l,j}} \times \frac{C_{l,SA}}{C_{SM,SA}} \quad (\text{Eq. 4})$$

274 where  $C_{SM,j}$  is the concentration of the target pollutant sorbed onto the sorbent material (mg g<sup>-1</sup>)  
 275 <sup>1</sup>),  $C_{l,j}$  is the concentration of the target pollutant in the liquid phase (mg L<sup>-1</sup>),  $C_{l,SA}$  is the  
 276 concentration of extracting agent in the liquid phase (g L<sup>-1</sup>),  $C_{SM,SA}$  is the concentration of  
 277 extracting agent onto the sorbent material (g g<sup>-1</sup>) and  $S$  is the selectivity ratio.

278 By using a configuration with a continuous flow through column, such adsorption  
 279 process leads to a much shorter exhausting time of extracting agents compared to breakthrough  
 280 time of target pollutants [70].

281  
 282 [Table 1 – Adsorption processes for the recovery of extracting agents – an overview of existing studies.](#)

Sorbent	Pollutants	Extracting agents	Configuration	References
Activated carbon	PHE <sup>1</sup>	TX100 <sup>2</sup>	Batch	[66]

Activated carbon	PHE <sup>1</sup> , FLUO <sup>3</sup> , BaA <sup>4</sup>	TX100 <sup>2</sup>	Column	[70]
Activated carbon	Trichloroethylene	HPCD <sup>5</sup>	Batch	[71]
Activated carbon	Mineral oil	RAMEB <sup>6</sup>	Column	[72]
Activated carbon	PHE <sup>1</sup>	Mixture SDS <sup>7</sup> -TX100 <sup>2</sup>	Batch	[73]
Activated carbon	PHE <sup>1</sup>	SDS <sup>7</sup>	Batch	[67]
Activated carbon	PHE <sup>1</sup>	TX100 <sup>2</sup>	Column	[74]
Activated carbon	FLUO <sup>2</sup>	TX100 <sup>2</sup>	Batch	[75]
Activated carbon	Chlorinated solvents	TX100 <sup>2</sup> , TX165 <sup>8</sup>	Column	[76]
Activated carbon	PHE <sup>1</sup>	TW40 <sup>9</sup> , TW80 <sup>10</sup> , B30 <sup>11</sup> , B35 <sup>12</sup>	Batch	[77]
Organo-bentonite	Various PAH <sup>13</sup>	TX114 <sup>14</sup> , TX100 <sup>2</sup> , TX305 <sup>15</sup>	Batch	[68]
Activated carbon	Hexachlorobenzene	Rhamnolipid	Column	[78]
Activated carbon	p-Cresol	TW80 <sup>10</sup>	Batch	[79]
Biochar	PHE <sup>1</sup> , FLUO <sup>3</sup> , PYR <sup>16</sup>	TX100 <sup>2</sup>	Batch	[80]
Activated carbon	PHE <sup>1</sup>	TW80 <sup>10</sup>	Batch	[81]
Activated carbon	Organochlorine pesticide	TX100 <sup>2</sup>	Batch	[82]
Macroporous resin SP850	PHE <sup>1</sup>	TX100 <sup>2</sup> , SDBS <sup>17</sup>	Batch	[83]
Organo-LDH <sup>18</sup>	PHE <sup>1</sup> , PYR <sup>16</sup>	SDBS <sup>17</sup> , B35 <sup>12</sup>	Batch	[69]

283

284 <sup>1</sup> Phenanthrene; <sup>2,8,14,15</sup> Triton X 100, 165, 114, 305; <sup>3</sup> Fluoranthene; <sup>4</sup> Benzo(a)anthracene; <sup>5</sup> HPCD <sup>6</sup> Randomly

285 methylated  $\beta$ -cyclodextrin; <sup>7</sup> Sodium dodecylsulfate; <sup>9,10</sup> Tween<sup>®</sup> 40, 80; <sup>11,12</sup> Brij<sup>®</sup> 30, 35; <sup>13</sup> Polycyclic aromatic

286 hydrocarbon; <sup>16</sup> Pyrene; <sup>17</sup> Sodium dodecylbenzene sulfonate; <sup>18</sup> Layered double hydroxides.

287



288           The selectivity of adsorption processes tightly relies on the reduction of competition  
289 phenomena for adsorption of target pollutants. Even if promising results have been obtained,  
290 further investigations are necessary [71]. Particularly, it is required to better understand  
291 competition mechanisms between partitioning of target pollutants inside micelles or inclusion  
292 complex and adsorption on the sorbent material [71].  $K_m$  and  $K_{CD}$  are important parameters to  
293 take into consideration for the availability of pollutants for adsorption [84]. Besides, other  
294 compounds from real SW solutions such as NOM could have strong adverse effects on the  
295 selectivity ratio. For example, selectivity ratio was divided by 2 in a fluoranthene/TX100  
296 mixture when adding 0.10 g L<sup>-1</sup> of fulvic acids because these compounds participate to  
297 additional pore blocking [75]. Besides, sand filtration is a required pre-treatment for real SW  
298 solutions in order to prevent too much rapid fouling of the activated carbon filter by particulate  
299 materials. The influence of the nature of EAs, target pollutants and sorbent materials (particle  
300 size, pore size distribution, etc.) should also be further investigated. The extracting agent and  
301 the sorbent material have to be carefully chosen in order to limit the phenomenon of pore  
302 blocking, which can lead to a reduction of active sites available for target pollutant adsorption.  
303 For example, it has been observed that the reduction of surface area of activated carbon after  
304 TX100 adsorption was mainly ascribed to the adsorption on micropores [67]. Pore size  
305 distribution could therefore be optimized in order to favour adsorption of target pollutants  
306 instead of extracting agents.

307           The regeneration of sorbent materials is also a crucial issue for the sustainability of  
308 adsorption processes. Thermal processes are currently widely used for activated carbon  
309 regeneration. When performing thermal desorption, a large amount of energy is consumed and  
310 a post-treatment of the contaminated gas stream is still required. Degradation of target  
311 pollutants can be achieved by heating at high temperature under oxidizing conditions, but such  
312 treatment usually reduce the potential for AC reuse because of a strong decrease of specific

313 surface area and adsorption capacity [85,86]. Therefore, alternative regeneration processes are  
314 currently investigated, such as biological treatments [87,88], microwave irradiation [70,89],  
315 ozone [86] or electro-Fenton (EF) processes [85,90,91]. The latter process seems to be a  
316 promising technique based on the use of activated carbon as cathode, which allows for both  
317 mineralization of organic pollutants and regeneration of the adsorption capacity of the spent  
318 activated carbon [91].

319

### 320 **III – Degradation processes**

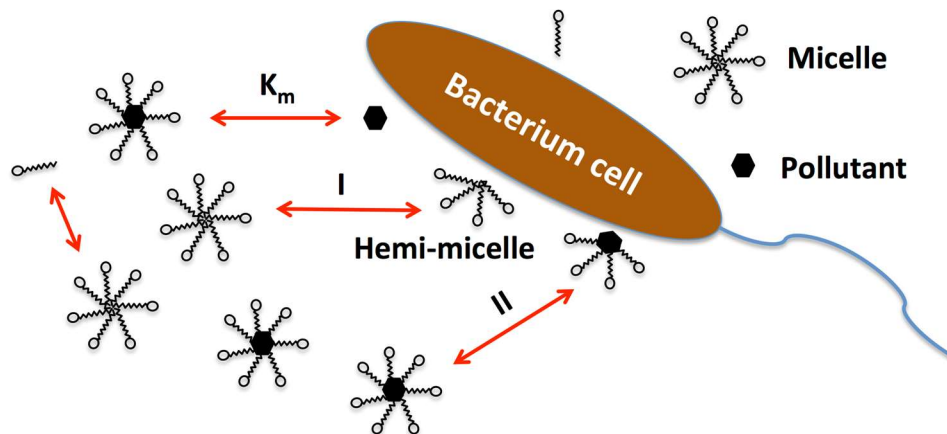
#### 321 **III.1 – Biological processes**

322 Biological treatment (mainly aerobic processes) is one of the most popular processes in  
323 water treatment for the removal of biodegradable organic compounds. It is based on the  
324 consumption of organic compounds by a large range of microorganisms. Recent progress  
325 allows the application of this kind of treatment to various effluents in terms of nature and  
326 organic load. For a specific compound, removal rates and pathways depend on several physical,  
327 chemical and microbiological aspects [1]. Some organic compounds can be recalcitrant to  
328 biodegradation for different reasons such as the absence of microorganism able to use them as  
329 substrate, the presence of toxic concentrations of organic compounds or the lower  
330 bioavailability due to the physicochemical parameters of the environment [92]. Therefore, the  
331 development of an adapted biomass to a specific SW solution and the influence of extracting  
332 agents on the bioavailability of target pollutants are two key parameters [93–96].

333 As the uptake of organic compounds by microorganisms occur mainly via the aqueous  
334 phase, partition of HOC in the CD inclusion complex or in the surfactant micellar pseudo-phase  
335 can strongly influence biodegradation kinetics [97–99]. Different mechanisms are actually  
336 involved in the biodegradation of target pollutants in surfactant-containing solutions [92]

337 (Figure 2). First, bacteria can directly utilize aqueous extra-micellar pollutants as substrate.  
 338 Then, pollutants diffusing from the micellar (or inclusion complex) to the aqueous phase  
 339 (kinetics in the range of microseconds to milliseconds) according to the micellar phase /  
 340 aqueous phase partition coefficient ( $K_m$ ) becomes also bioavailable. Biodegradation of  
 341 pollutants solubilized in micelles can also occur due to the formation of a hemi-micellar layer  
 342 around the bacteria cells (step I, Figure 2) and transfer of pollutants from the micellar phase to  
 343 this hemi-micellar layer (step II, Figure 2) [92]. However, these two steps might be kinetically  
 344 limiting for biodegradation [92].

345



346

347 Figure 2 – Organic pollutant uptake by bacteria in soil washing solutions.  $K_m$  is the micellar phase /  
 348 aqueous phase partition coefficient of the target pollutant. I: equilibrium between the hemi-micellar layer  
 349 around the bacteria cells and micelles in the aqueous phase. II: equilibrium between pollutants  
 350 solubilized in hemi-micelles around the bacteria cells and micelles in the aqueous phase.

351

352

353 The complexity of such system often led to contradictory results [92]. On the one hand,  
 354 the enhancement of HOC biodegradation in solutions containing extracting agents has been  
 355 mainly attributed to the increase of the solubility and bioavailability of HOCs [94,95,100,101].  
 356 On the other hand, adverse effects have been often ascribed to the inhibition of the direct contact  
 357 between microbial cells and target pollutants [97,99,102], toxicity of extracting agents towards

358 microorganisms [102–104] or preferential uptake of extracting agents as substrate  
359 [103,105,106]. Overall, the following trend is often observed the presence of low concentration  
360 of extracting agent (below or close to the CMC for surfactants) enhances HOC biodegradation,  
361 while bioavailability and biodegradability are reduced at high concentration of extracting agent  
362 [92,96,98,99].

363 As extracting agents are usually used at high concentration during SW processes, it is  
364 very difficult to achieve a selective biodegradation of target pollutants in SW solutions because  
365 of the protective environment formed by extracting agents. Therefore, only few authors have  
366 investigated SW solution recirculation after biological treatment. Navarro et al. [96] observed  
367 a selective biodegradation of PYR from a SW solution containing deoxyribonucleic acid (DNA)  
368 as extracting agent. The removal of PYR reached 99% after 1 d, while more than 98% of  
369 deoxyribonucleic acid was recovered. This result was explained by the stability of DNA against  
370 the aerobic bacteria (*Sphingomonas* sp.) used in this study for degradation of PYR. Thus, the  
371 treated solution was re-used for four SW cycles with synthetic contaminated soil, with a high  
372 extraction capacity. Recently, it was also reported that the use of a bacterial consortium could  
373 allow the reuse of a biologically-treated SW solution containing PHE and TW80 as extracting  
374 agent [107].

375

## 376 **III.2 – Chemical, photochemical and electrochemical advanced oxidation processes**

377

### 378 **III.2.1 – Removal of target pollutants: generation of oxidant species and reaction**

#### 379 **mechanisms**

380 Advanced oxidation processes (AOPs) have been largely studied for the treatment of  
381 SW solutions. AOPs are well-known for the removal of recalcitrant organic pollutants [108].  
382 They are based on the *in situ* production of strong oxidants, mainly hydroxyl radicals, the

383 second most powerful oxidizing agent known ( $E^\circ(\cdot\text{OH}/\text{H}_2\text{O}) = 2.8 \text{ V/SHE}$ ) [109]. Hydroxyl  
384 radicals are characterized by a very short lifetime (in the range of nanoseconds) and a non-  
385 selective feature towards organic compounds [110]. There are four main reaction pathways for  
386 the reaction of hydroxyl radical with organic compounds, including hydrogen atom abstraction  
387 (dehydrogenation), addition to an unsaturated bond (hydroxylation), ipso substitution  
388 (hydroxylation with release of halogen ion) and electron transfer (redox reactions) [110,111].  
389 Aromatic compounds are the most reactive species with hydroxyl radicals since hydroxylation  
390 is typical for these kinds of compounds with rate constants in the range  $10^8 - 10^{10} \text{ M}^{-1} \text{ s}^{-1}$ , while  
391 lower values are reported ( $10^6 - 10^8 \text{ M}^{-1} \text{ s}^{-1}$ ) for alkanes and alcohols for which reaction occurs  
392 through dehydrogenation [110]. Different methods are used for the production of hydroxyl  
393 radicals. Mainly photochemical, electrochemical, and Fenton-based processes have been  
394 applied to the treatment of SW solutions [1,18,25,112-118].

395 Heterogeneous photocatalysis is based on photoactivation of a semiconductor (e.g.  
396  $\text{TiO}_2$ ), leading to the production of an electron in the conduction band and a positive hole in the  
397 valence band [119,120]. These materials have a void energy region avoiding the recombination  
398 of electrons and holes. Thus, the reaction of electrons and positive holes with  $\text{O}_2$ ,  $\text{H}_2\text{O}$ , and  $\text{OH}^-$   
399 leads to the production of oxidant species on the surface of the photocatalyst and in the bulk,  
400 including  $\cdot\text{OH}$ ,  $\text{H}_2\text{O}_2$ ,  $\text{O}_2^-$  [121].

401 The production of hydroxyl radicals during Fenton-based processes comes from the  
402 catalytic decomposition of hydrogen peroxide by ferrous iron [108,110]. The effectiveness of  
403 the process can be enhanced by using UV irradiation (photo-Fenton based processes) [122,123],  
404 allowing a better regeneration of ferrous iron and the production of additional hydroxyl radicals  
405 from the photolysis of  $\text{Fe}(\text{OH})^{2+}$  [124]. The development of iron-containing solids catalysts is  
406 also a promising way for reducing the production of iron sludge and allowing the application  
407 of the process at circumneutral pH [125,126].

408 Electrochemical processes can lead to the production of hydroxyl radicals from (i)  
409 anodic oxidation by water discharge on the surface of anode with high oxygen evolution  
410 overpotential (such as boron-doped diamond, BDD) [127,128], (ii) EF process through *in situ*  
411 production of the Fenton's reagent [110]: H<sub>2</sub>O<sub>2</sub> is produced from the two electron reduction of  
412 oxygen at a suitable cathode (such as gas diffusion or three-dimensional electrodes using  
413 carbon-based porous materials combined with injection of compressed air in the solution),  
414 while only a catalytic amount of ferrous iron is supplied and continuously regenerated by  
415 reduction of ferric iron at the cathode [129]. The EF process allows increasing the efficiency of  
416 the Fenton's process by avoiding the addition of chemical reagents, reducing waste reactions  
417 and avoiding sludge formation [1,130]. Oxidation of inorganic anions in the solution (SO<sub>4</sub><sup>2-</sup>,  
418 Cl<sup>-</sup>, etc.) can also lead to the homogeneous production of oxidant species in the bulk such as  
419 active chlorine, persulfate, sulphate radical, which also participate to the oxidation of organics  
420 ("mediated oxidation") [131,132].

421 Recently, a great attention has also been given to the development of AOPs based on  
422 persulfate activation, due to several inherent advantages of sulphate radicals such as (i) the  
423 convenience of storage and transportation of solid persulfate, (ii) the different way to activate  
424 persulfate (e.g. thermal or electrochemical processes, Fe<sup>2+</sup>) and (iii) the longer lifetime of  
425 sulphate radicals compared to hydroxyl radicals [133–137].

426 Total degradation of target pollutants as well as high mineralization rate of SW solutions  
427 is usually achieved when using such AOPs. Some examples of degradation kinetic rates are  
428 reported in Table 2. When optimized operating conditions are used, the apparent degradation  
429 kinetic rates of target pollutants in SW solutions are usually in the range 0.5 – 5 h<sup>-1</sup>.

430  
431 **Table 2 – Degradation kinetic rates of target pollutants in soil washing solution by advanced**  
432 **oxidation processes**

Process	Pollutants (mM)	Extracting agent (mM)	Maximum degradation kinetic rate (h <sup>-1</sup> )	Reference
Photocatalysis (TiO <sub>2</sub> )	NAP <sup>1</sup> (0.03)	B35 <sup>2</sup> (1.2)	2.04	[138]
Photocatalysis (TiO <sub>2</sub> )	PCP <sup>3</sup> (0.2)	β-CD <sup>4</sup> , MCD <sup>5</sup> , HPCD <sup>6</sup> (0 – 5)	5.3	[139]
Solar-Photocatalysis (TiO <sub>2</sub> )	NAP <sup>1</sup> (0.2)	TX100 <sup>7</sup> (0.04 – 0.13)	4.2	[140]
Photocatalysis (TiO <sub>2</sub> )	Aromatic compounds	B35 <sup>2</sup> (25)	0.12 – 3.48	[141]
Photocatalysis (TiO <sub>2</sub> )	PCP <sup>3</sup> (0.018)	TX100 <sup>7</sup> (0.023 – 1.01)	3.3	[89]
Solar-Photocatalysis (TiO <sub>2</sub> )	Alkylphenols	B35 <sup>2</sup> , C <sub>12</sub> E <sub>8</sub> , SDS <sup>8</sup> , mixtures (1 – 15)	9.3	[20]
Photo-Fenton	TNT <sup>9</sup> (0.5)	None (water)	4.86	[142]
		MCD <sup>5</sup> (5)	10.05	
EF	PCP <sup>3</sup> (0.1)	HPCD <sup>6</sup> (5)	0.546	[143]
EF	PHE <sup>10</sup> (0.1)	HPCD <sup>6</sup> (8.8)	1.56	[144]
		TW80 <sup>11</sup> (0.6)	0.78	
EF	PAHs	HPCD <sup>6</sup> (6)	0.83 – 2.61	[145]
		TW80 <sup>11</sup> (5.6)	0.20 – 0.72	
Anodic oxidation	NAP <sup>1</sup> (0.83)		0.9	[146]
	PYR <sup>12</sup> (0.27)	CAS <sup>13</sup> (9.1)	0.66	

433

434 <sup>1</sup> Naphthalene; <sup>2</sup> Brij<sup>®</sup> 35; <sup>3</sup> Pentachlorophenol; <sup>4</sup> β-cyclodextrin; <sup>5</sup> Methyl-β-cyclodextrin; <sup>6</sup> Hydroxypropyl-β-

435 cyclodextrin; <sup>7</sup> Triton X 100; <sup>8</sup> Sodium dodecylsulfate; <sup>9</sup> Trinitrotoluene; <sup>10</sup> Phenanthrene; <sup>11</sup> Tween<sup>®</sup> 80; <sup>12</sup>

436 Pyrene; <sup>13</sup> Cocamidopropylhydroxysultaine

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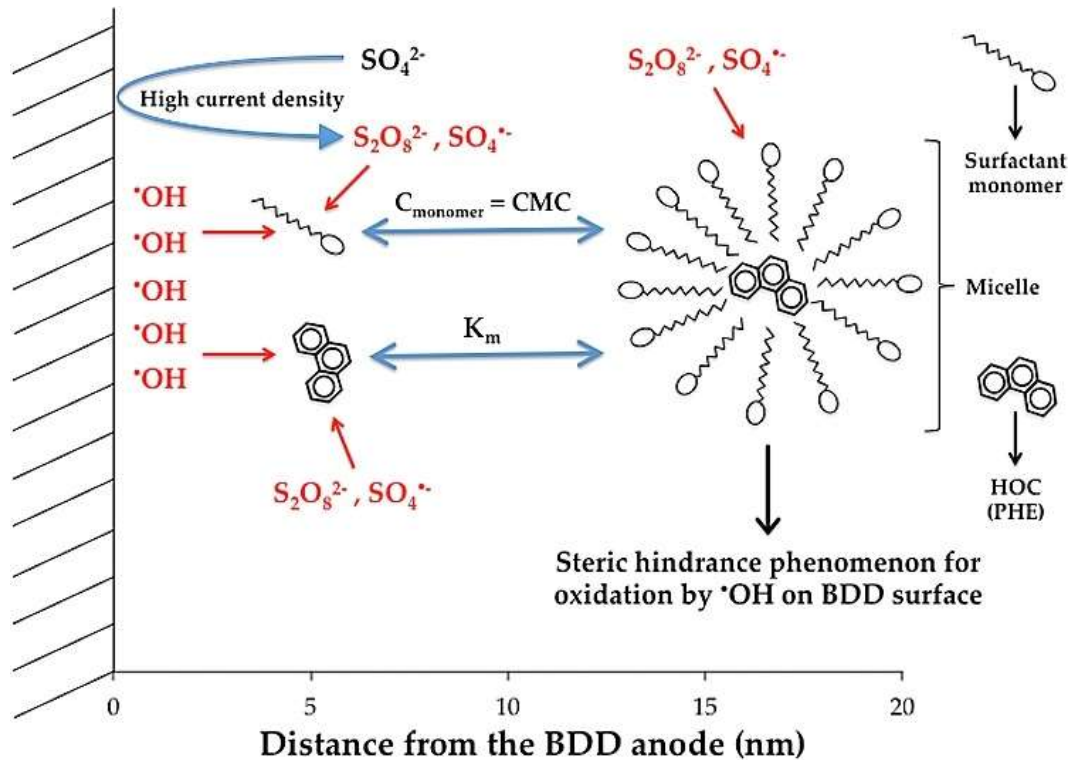
438 The specificity of SW solutions requires giving a special attention for the understanding  
439 of oxidation mechanisms, according to the process used, operating conditions, target pollutant  
440 and extracting agent used. Target pollutant removal usually requires high energy and/or reagent  
441 consumption, due to the high organic load of SW solutions. Concentrations of extracting agents  
442 are usually in the range of 1-100 g L<sup>-1</sup> [1,11]. Moreover, a large amount of soil organic matter  
443 is also mobilized in the SW solution [1]. Since hydroxyl radicals act in a non-selective way,  
444 there is therefore a huge competition for oxidation [112,147–149] and wasting reactions from  
445 hydroxyl radical scavengers strongly reduce the kinetics of target pollutant degradation. This  
446 high oxidant demand is the main issue for the treatment of SW solution by AOPs. For example,  
447 49 g L<sup>-1</sup> (using sequential addition) and 30 g L<sup>-1</sup> of H<sub>2</sub>O<sub>2</sub> was necessary to achieve 95% and  
448 96% COD removal from a SW solution [25,146]. As regards to the EF process, the treatment  
449 of a SW solution containing PAH and TW80 as EA, required 0.69 kWh per gram of TOC  
450 removed [145].

451 Besides, solubilization of target pollutants inside surfactant micelles and inclusion  
452 complex with CDs reduces the effectiveness of oxidation processes by decreasing the  
453 availability of target pollutants towards oxidant species in the aqueous phase because of  
454 formation of a protective environment [132,144,150,151]. However, different mechanisms  
455 occur when CDs are used as extracting agents during Fenton-based processes. The formation  
456 of a ternary complex CD-pollutant-Fe<sup>2+</sup> has been observed [152,153]. This phenomenon allows  
457 improving the effectiveness of Fenton-based processes thanks to the production of hydroxyl  
458 radicals close to target pollutants [142,144,154].

459 As regards to anodic oxidation, hydroxyl radicals are only present in a thin layer (few  
460 nm) on the anode surface. Therefore, reaction with organic compounds strongly depends on  
461 their mass transport from the bulk to the anode surface [128]. Formation of large size micelles  
462 was reported to prevent oxidation on the anode surface, due to a phenomenon of steric hindrance



463 [155]. However, production of oxidant species in the bulk through mediated oxidation  
 464 (favoured at high current density) can still participate in the degradation of micelles during  
 465 electro-oxidation of SW solutions [132] (Figure 3).



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Figure 3 – Schematic view of oxidation mechanisms occurring during the treatment of a soil washing solution (containing phenanthrene (PHE) as hydrophobic organic compound (HOC)) by anodic oxidation using boron-doped (BDD) anode. Reprinted from [23].

Mechanisms of organic compounds removal from SW solutions during photocatalytic processes also strongly depend on extracting agent characteristics and concentration. Extracting agents influence the adsorption of pollutants onto photocatalysts and thus, the availability of pollutants for oxidant species [1]. Improved photocatalytic degradation of HOCs was observed when using concentrations of surfactant below or close to the CMC [150]. Combination of surfactant monomers with the surface of TiO<sub>2</sub> by hydrophobic (non-ionic surfactants) or electrostatic (cationic surfactants) interactions was reported to form a superficial hydrophobic reactive monolayer in which HOCs have high affinity [150,151,156]. Less favourable effects

479 were observed for photocatalytic degradation of more hydrophilic compounds such as oxidation  
480 by-products [151]. It was attributed to the decrease of their availability for oxidant species  
481 because of the lower fraction of organic compounds implanted in the superficial hydrophobic  
482 reactive monolayer (lower  $K_m$ ) and/or a different locus of implementation of these organic  
483 compounds inside this monolayer (closer to the hydrophilic heads of surfactants) (Figure 4). At  
484 high surfactant concentration, photocatalytic degradation of HOCs strongly decreases due to  
485 the hydroxyl radical scavengers as well as competitive partition of HOCs within micelles in the  
486 bulk (that prevent reaction at the surface of the photocatalyst). Competition between surfactants  
487 and HOCs for the occupation of active sites on the catalyst surface may also have strong adverse  
488 effects [150,151,157]. Similar effects of concentration-dependant inhibition have been  
489 observed when using CDs as extracting agent. Low concentration of CDs promotes the  
490 photocatalytic degradation of HOC having high inclusion constant with CDs [158–160], while  
491 high CD concentration hinders target pollutant degradation [62,139]. Unfortunately, SW  
492 solutions usually contain high concentration of extracting agent. Interestingly, a lower  
493 inhibitory effect was observed when using surfactants with bigger hydrated polar head, due to  
494 the reduction of surfactant adsorption at the surface of the catalyst [18,112].  
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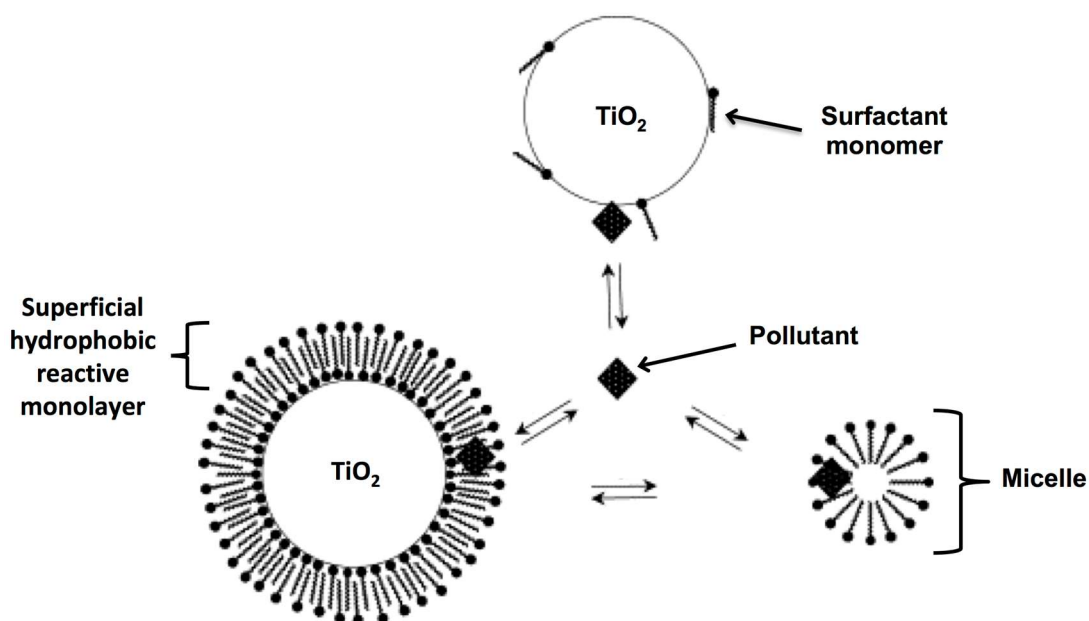


Figure 4 –Partition equilibria of target pollutants during the treatment of a SW solution by a  $\text{TiO}_2$ -based photocatalytic process. Reprinted from [1].

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Some other non-AOPs chemical oxidation processes have also been applied to the treatment of SW solution, particularly ozone, activated persulfate [135,136,161,162], and electrochemical processes using active electrodes that do not produce hydroxyl radicals [128,163]. Lower effectiveness for the removal of target pollutants has been reported because of the generation of weaker oxidant species [1]. For example, degradation kinetic rates observed for the removal of PAH from SW solutions by electro-oxidation using graphite electrodes were in the range 0.02-0.2  $\text{h}^{-1}$  [21,164]. As regards to ozone, it was observed significant removal of PAH and chlorophenols from SW solutions (45-65% removal) only when using high doses ( $> 500 \text{ mg O}_3 \text{ L}^{-1}$ ) [165,166]. Besides, it was reported that high concentration of surfactant decreases gas-liquid mass transfer for both (semi)-volatile pollutants and ozone, thus reducing the effectiveness of the ozone process [167].

### III.2.2 – Effectiveness for the recovery of extracting agents

513 SW solution reuse by implementing a degradation process means that the aim is to  
514 achieve both maximum removal of target pollutants and minimum degradation of extracting  
515 agents [1]. Thus, the treated SW solution might keep similar extraction capacity than a fresh  
516 SW solution without addition of extracting agents. However, two main issues are involved: (i)  
517 the protective environment for target pollutants formed by surfactant micelles and CDs, which  
518 usually decreases the availability of target pollutants for degradation ( $K_m$  and  $K_{CD}$  are crucial  
519 parameters), (ii) the degradation of organic compounds in a non-selective way [1].

520 Several authors have also investigated the possibility to recover extracting agents by  
521 optimizing operating conditions of AOPs in order to promote the degradation of target  
522 pollutants and decrease the degradation rate of extracting agents. Even if hydroxyl radicals are  
523 considered as non-selective oxidants, different reaction kinetics with target pollutants and other  
524 extracting agents are obtained according to their chemical structure. For example,  
525 perfluorinated surfactants present the interesting characteristic to be highly recalcitrant to  
526 oxidation by hydroxyl radicals, however, they are considered as toxic persistent organic  
527 pollutants and they cannot be used for soil remediation by SW [168]. Aromatic compounds are  
528 also considered as highly reactive species [110]. A slower degradation rate of extracting agents  
529 compared to aromatic target pollutants can be used for partial recovery of extracting agents. As  
530 complete mineralization of target pollutants involves longer treatment time and higher  
531 degradation rate of extracting agents [1], only degradation of pollutants is usually considered.  
532 Therefore, the toxicity of degradation by-products [169,170] should also be assessed. Mousset  
533 et al. [145] observed that a second SW step with a partially oxidized SW solution (containing  
534 PAHs and TW80 as EA) by the EF process did not affect negatively the soil microbial activity,  
535 compared to the use of a fresh SW solution. This result was ascribed to the production of  
536 hydroxylated by-products with higher biodegradability and solubility than initial compounds  
537 [145,171].

538 As regards to Fenton-based processes, promising results have been observed by using  
539 CDs as extracting agents. The formation of a ternary complex CD-pollutant-Fe<sup>2+</sup> allows  
540 improving the availability of target pollutants towards hydroxyl radicals and higher degradation  
541 kinetics of target pollutants were achieved compared to CDs [144,154]. By using the EF process,  
542 better selective degradation was also achieved when using HPCD instead of TW80 as extracting  
543 agent [144].

544 During photocatalytic processes, the formation of micelles in the bulk reduces the  
545 availability of surfactants towards oxidant species generated on the photocatalyst surface  
546 [150,151,157] (Figure 4). Even if it might be considered as an interesting behaviour for  
547 minimizing the degradation rates of EAs, it was observed to do not improve the selectivity of  
548 the process. In fact, much lower degradation kinetic of target pollutants is usually also observed  
549 because of the partitioning within micelles. Thus, it was actually reported higher rate of  
550 recovery of TX100 after full removal of a target pollutant (pentachlorophenol) when a low  
551 concentration of TX100 was used [150].

552 Processes based on the activation of persulfates using iron ions or thermal activation  
553 have also been recently developed for the treatment of SW solution. Compared to hydroxyl  
554 radicals, sulfate radicals present some more selective features that might be useful for achieving  
555 selective degradation of target pollutants [136,172].

556 During anodic oxidation processes, it has been reported that low current density strongly  
557 hinders degradation of extracting agents due to a steric hindrance phenomenon for oxidation of  
558 micelles on the anode surface. Lower degradation of extracting agents also results from the  
559 decrease of mediated oxidation in the bulk at low current density [23,155]. Thus, free extra-  
560 micellar target pollutants, continuously released in the solution (according to  $K_m$ ) can be more  
561 selectively degraded [23] (Figure 3).

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Table 3 – Promising results reported in the literature on the recovery of extracting agents by using degradation processes

Process	Pollutants (P) (mg L <sup>-1</sup> )	Extracting agent (EA) (g L <sup>-1</sup> )	Effectiveness	Ref
Photocatalysis	Dibenzothiophene (0.3 – 1.3)	TX100 <sup>1</sup> (0.14 – 0.25)	Full degradation of P / none degradation of EA	[173]
EF	PHE <sup>2</sup> (17.8)	HPCD <sup>3</sup> (10)	Full degradation of P / 10% degradation of EA	[144]
Electro-oxidation (graphite electrodes)	PHE <sup>2</sup> (35)	TW80 <sup>4</sup> (10)	95% removal of P / Similar extraction capacity of the treated solution	[174]
Electro-oxidation (graphite electrodes)	PHE <sup>2</sup>	HPCD <sup>3</sup> (1.0)	Full removal of P / Similar extraction capacity of the treated solution	[164]
Fenton	p-Cresol (20)	TW80 <sup>4</sup> (0.86)	Full degradation of P / 10% degradation of EA	[175]
Activated persulfate	Nitrobenzene	SDBS <sup>5</sup> (8.2)	Better selective degradation than with the Fenton process	[161]
Anodic oxidation (BDD <sup>6</sup> anode)	PAHs from real SW solution (1 – 5)	TW80 <sup>4</sup> (6.6)	75-90% removal of P / Similar extraction capacity of the treated solution	[23]
Activated persulfate	PHE <sup>2</sup> (10)	TW80 <sup>4</sup> (10)	96% removal of P / 20% removal of EA	[176]
Fenton	COCs <sup>7</sup> from real SF solution (3700)	E-Mulse 3 <sup>®</sup> (11)	>80% removal of P / Similar extraction capacity of the treated solution	[177]

565 <sup>1</sup> Triton X 100; <sup>2</sup> Phenanthrene; <sup>3</sup> Hydroxypropyl-  $\beta$  -cyclodextrin; <sup>4</sup> Tween<sup>®</sup> 80; <sup>5</sup> Sodium  
566 dodecylbenzenesulfonate; <sup>6</sup> Boron-doped diamond; <sup>7</sup> Chlorinated organic compounds

567

568 However, contradictory results are also observed and the details of mechanisms leading  
569 to the selective removal of pollutants solubilized within micelles are not fully understood.  
570 Further development of selective degradation processes depends on the better understanding  
571 and control of degradation mechanisms and kinetics, for which the nature and concentration of  
572 EAs, target pollutants, and oxidant species have a crucial effect [172].

573 It is also worth noting that the critical parameter is not the amount of extracting agent  
574 recovered but the extraction power of the treated solution. Important differences are usually  
575 observed because of analysis artefacts of extracting agents, generation of degradation by-  
576 products still solubilized in micelles and production of extracting agent by-products with  
577 extracting properties [1]. For example, it was observed that a partially oxidized solution (by  
578 using the EF process) containing 11% less of HPCD than the initial solution achieved slightly  
579 higher PAH removal from soil than a fresh HPCD solution [145]. This result was ascribed to  
580 the extraction capacity of hydroxylated HPCD (degradation by-products) with an internal  
581 hydrophobic space that remained intact. Interestingly, [Dominguez et al. \[177\]](#) also reported that  
582 Fenton oxidation of a SF solution led to >99% degradation of an additive of a commercial  
583 extracting agent (E-Mulse 3<sup>®</sup>), however, the equivalent surfactant concentration was only  
584 decreased by 40-50%, the interfacial tension of the solution was not altered and the extraction  
585 capacity of the solution remained constant. On the contrary, the reduction of 21% of the TW80  
586 concentration during the EF treatment of a SW solution strongly decreased the extraction power  
587 of the partially oxidized solution compared to a fresh TW80 solution [145]. Such results might  
588 be ascribed to the artefacts in analysis of extracting agents (detection of degradation by-  
589 products identified as TW80 while they have lower extraction capacity).

590

### 591 **III.3 - Soil washing solution reuse or total removal of the organic load? The example of** 592 **the anodic oxidation process**

593 Recently, two different treatment strategies have been identified for the treatment of SW  
594 solutions by anodic oxidation [22,23]. First, it was observed that treating a real SW solution  
595 (with TW80 as EA) by anodic oxidation at low current density (1.8 mA cm<sup>-2</sup>) during a long  
596 treatment time (23 h) lead to the selective degradation of PAHs as target pollutants and allow  
597 the recovery of the extraction capacity of the treated SW solution [23]. It was also emphasized

598 that a high surfactant concentration improves the selective degradation of target pollutants with  
599 only a slight decrease of degradation kinetics of PAHs. Therefore, high concentration of  
600 surfactant might be used in order to increase the transfer of PAHs from the soil-sorbed fraction  
601 to the washing solution. In fact, the consumption of an additional amount of surfactant might  
602 be compensated by the high rate of recovery of extracting agents during the treatment by anodic  
603 oxidation at low current density. A second treatment strategy has been reported for the removal  
604 of the organic load of a synthetic SW solution containing PHE as target pollutant and TW80 as  
605 extracting agent [22]. It is based on the combination of anodic oxidation with a biological  
606 treatment.

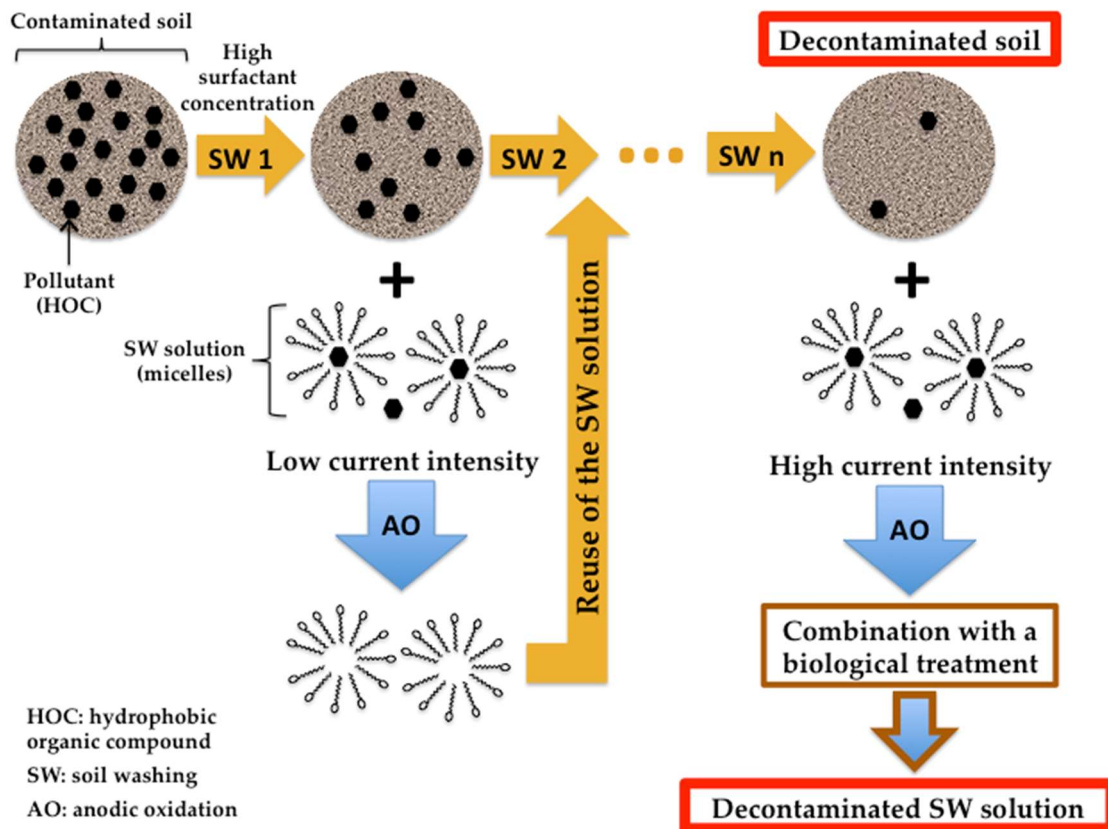
607         It might be actually interesting to use these two different treatment strategies in a  
608 complementary way. A crucial advantage of anodic oxidation is the possibility to easily manage  
609 the operating conditions, particularly the current density [178]. Thus, an anodic oxidation  
610 treatment system might be able to implement both strategies (Figure 5). First, low current  
611 density might be initially used for the reuse of the SW solution. Then, when sufficient pollutant  
612 removal rate from soil would be achieved, the same system might treat the SW solution at high  
613 current density in order to remove the organic load with a possible combination with a  
614 biological treatment.

615         However, a systematic analysis taking into consideration all the different parameters of  
616 such complex environmental engineering issue would be required for concluding on the best  
617 treatment strategy (e.g. using life cycle assessment). From data obtained, only a short cost-  
618 benefit analysis can be performed on energy consumption, which is a critical parameter during  
619 anodic oxidation. It was reported that SW solution treatment at low current density only  
620 required 13.2 kWh m<sup>-3</sup> for the reuse of the treated solution [23]. By considering the price of the  
621 electricity as 0.12 € kWh<sup>-1</sup> (in France), it corresponds to an energy cost of 1.6 € m<sup>-3</sup> of SW  
622 solution treated. Considering a price for TW80 at 4.0 € kg<sup>-1</sup>, it means that 1 m<sup>3</sup> of a solution at



623 5.0 mM of TW80 ( $6.6 \text{ g L}^{-1}$ ) would cost 26 €. Therefore, the saving arising from the reuse of  
 624 the SW solution may compensate costs related to energy consumption of anodic oxidation. For  
 625 comparison, the energy consumption was 39 kWh per kg of COD removed when using anodic  
 626 oxidation at high current density for the removal of the organic load. By considering 3 h of pre-  
 627 treatment (29% COD removal of an initial solution at  $2.7 \text{ g L}^{-1}$ ), it corresponds to a consumption  
 628 of  $30.5 \text{ kWh m}^{-3}$  ( $3.7 \text{ € m}^{-3}$ ) [22]. This value is much higher than the energy consumption  
 629 required for SW solution reuse. Moreover, the energy consumption of the post-biological  
 630 treatment are not considered. However, a final step for the removal of the organic load from  
 631 SW solutions would be always required prior discharge in order to avoid any contamination of  
 632 the aquatic environment.

633



634

635 **Figure 5** - Treatment strategy for soil remediation using soil washing and anodic oxidation processes.

636 Anodic oxidation at low current intensity allows the reuse of the soil washing solution for n soil washing

637 steps. Anodic oxidation at high current intensity combined with a biological treatment allows the removal  
638 of the organic load for final disposal of the soil washing solution.

639

640

#### 641 **IV – Discussion**

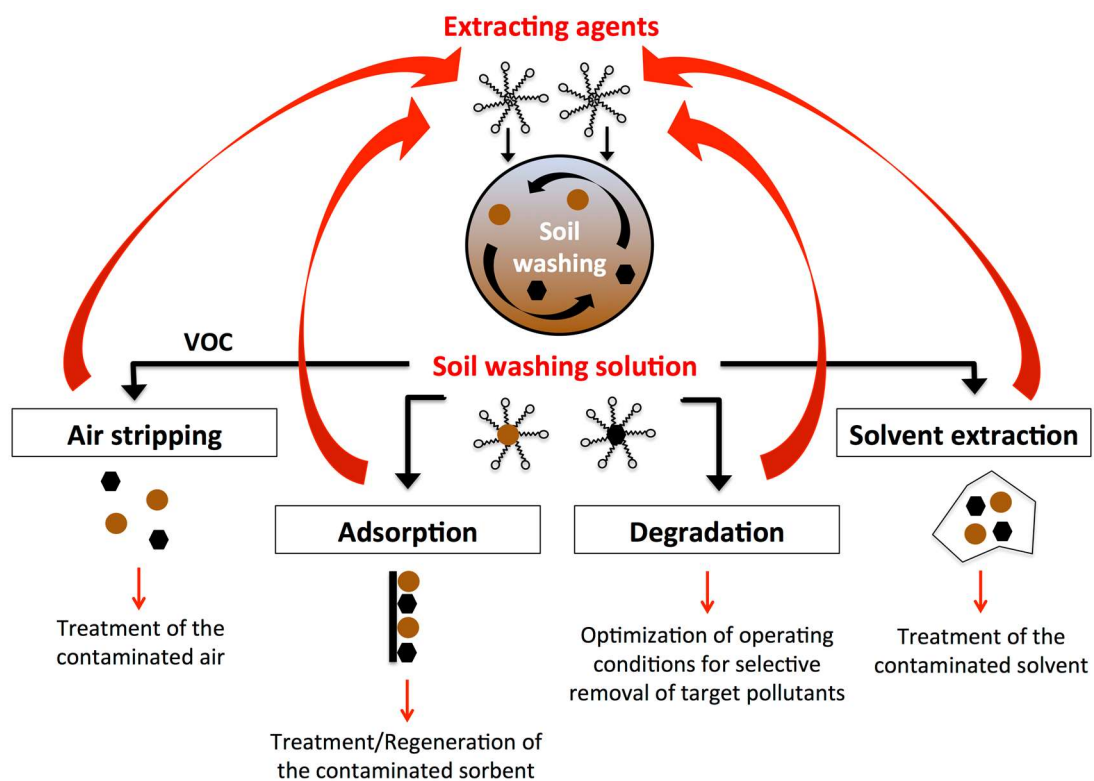
642 Several technologies have been critically reviewed in this article (Figure 6). The choice  
643 of the most suitable technique depends strongly on characteristics of the SW solution (Table 3).  
644 Air stripping and related technologies (such as vacuum extraction or pervaporation) allows the  
645 effective and selective removal of compounds such as trichloroethylene or perchloroethylene  
646 from SW solutions. The choice of an adapted organic solvent for a liquid-liquid extraction can  
647 lead to an effective extraction of HOCs in the organic phase while a large amount of extracting  
648 agents is also recovered. The first drawback is that foaming issues have to be managed. The  
649 disposal of the contaminated solvent also represents a critical drawback for the sustainability  
650 of the process. Besides, the adsorption process appeared as a promising process for the selective  
651 separation of target pollutants from SW solutions. Whatever the separation process used, high  
652 concentrations of extracting agent usually reduce separation effectiveness due to the partition  
653 of target pollutants in the micellar pseudo-phase or inclusion complex with CDs ( $K_m$  or  $K_{CD}$  are  
654 crucial parameters). Therefore, the optimal conditions for SW (high concentration of extracting  
655 agent and high  $K_m$  or  $K_{CD}$ ) have adverse effects on selective separation processes. Furthermore,  
656 a post-treatment of the contaminated air/sorbent/solvent is required, thus increasing the global  
657 cost of the whole treatment strategy.

658 Optimization of operating parameters for degradation processes can also lead to higher  
659 degradation rates of target pollutants compared to extracting agents. These results are mainly  
660 ascribed to the different reactivity of target pollutants and extracting agents with oxidant species.

661 The heterogeneous production of hydroxyl radicals at the anode surface during the anodic

662 oxidation process was also reported to promote selective oxidation of target pollutants when  
 663 using low current density. Compared to separation process, a lower amount of extracting agents  
 664 is usually recovered by using degradation processes but the great advantage lies in the  
 665 simultaneous degradation of target pollutants. In case of partial recovery of EAs, periodic re-  
 666 injection of fresh extracting agents might be considered for maintaining the extraction capacity  
 667 of the reused solution.

668



669

670 Figure 6 – Treatment strategies for the recovery of extracting agents from contaminated soil washing  
 671 solutions.

672

673 Table 3 – Important operating conditions, target pollutant and extracting agent characteristics to  
 674 consider according to the choice of the process used. CMC: critical micelle concentration;  $K_m$ : micellar  
 675 phase / aqueous phase partition coefficient;  $K_{CD}$ : complex stability (or equilibrium) constant between  
 676 organic compounds and cyclodextrins

Process	Important target pollutant characteristics (optimal*)	Important extracting agent characteristics (optimal*)	Important operating conditions
Air stripping	Henry constant (low), saturated vapor pressure (high), $K_m/K_{CD}$ (low)	Henry constant (high), concentration (low), $K_m/K_{CD}$ (low)	Limitation of foaming (hollow fiber membranes, vacuum processes)
Liquid-liquid extraction	Solvent-water partition coefficient (high), $K_m/K_{CD}$ (low)	Solvent-water partition coefficient (low), concentration (low), $K_m/K_{CD}$ (low)	Limitation of emulsification and amount of solvent used (hollow fiber membranes), highly hydrophobic solvent
Membrane processes	Oil-containing mixture (for selective removal), $K_m$	Concentration, $K_m$ , micelle size, CMC, effect on concentration polarization	Ultrafiltration (for micellar-enhanced ultrafiltration), fouling control (depending on fouling materials)
Adsorption processes	Partition coefficient on the sorbent material (high), $K_m/K_{CD}$ (low)	Partition coefficient on the sorbent material (low), concentration, $K_m/K_{CD}$ (low)	Sorbent material (pore size distribution) and dose
Degradation processes	Reactivity (high), $K_m/K_{CD}$	Reactivity (low), concentration, nature, $K_m/K_{CD}$	Optimized for selective degradation of target pollutants

677 \* Optimal conditions for the selective removal of target pollutants from SW solutions (different from optimal  
678 conditions for the SW step)

679

680 Further studies on real contaminated soils are required in order to assess the efficiency  
681 of these processes at real field scale. The complex mixture of compounds in real SW solutions  
682 involves strong competition mechanisms, which often reduce separation and degradation  
683 effectiveness. Several studies are performed on synthetic SW solutions, without taking into  
684 consideration the influence of clay and silt particles. Additional studies would be required in  
685 order to a better understand the impact of these compounds on selective separation or  
686 degradation processes. The extraction capacity of fresh and recovered SW solutions should also  
687 be always assessed with real contaminated soils. The use of artificial or spiked soils usually  
688 leads to a strong overestimation of the efficiency due to a lower sequestration of pollutants. For

689 example, it has been observed that ageing and weathering of tar oil-contaminated soil leads to  
690 the formation of resinated materials which strongly reduce mass transfer of PAHs from soil to  
691 the washing solution [10,189]. Finally, there is an important lack of data in the scientific  
692 literature on pilot or field scale applications in this context of most of processes (particularly  
693 selective degradation and adsorption processes). These data will be crucial for assessing the  
694 real potential of these treatment strategies.

695 Nature and concentration of the extracting agent used during the SW step are also  
696 critical parameters for the treatment of SW solutions (Table 3). Thus, the implementation of  
697 optimal operating conditions for SW should also take into consideration the impact of the nature  
698 and concentration of the extracting agent chosen on the treatment of the SW solution.  
699 Particularly, its capacity to be recovered and reused for several SW steps should be assessed in  
700 order to improve the sustainability of the whole process. Optimizing the recovery of extracting  
701 agents requires taking into consideration all the steps involved in the soil remediation process  
702 (Figure 1).

703

## 704 **V – Conclusions and Recommendations**

705 EA-enhanced SW is an effective treatment strategy for the removal of organic pollutants  
706 when it is applied to a suitable contaminated soil. The optimization of operating parameters  
707 (nature of the EA, concentration and other operating conditions) is the most direct way to save  
708 EAs, improve extraction effectiveness and implement a cost-effective process. However, an  
709 appropriate treatment strategy of the SW solution allowing the recovery of extracting agents  
710 and the reuse of the SW solution is also a crucial step in order to save large amounts of  
711 extracting agents and to improve the sustainability and ecological footprint of the whole  
712 remediation process.

713 Promising results have been reported for the recovery of extracting agents from SW  
714 solutions but further studies are necessary in order to assess the robustness of each process and  
715 the possibility for scaling up. Then, the choice of the most suitable technology (and operating  
716 conditions) depends on the physicochemical characteristics of each specific SW solution. It is  
717 therefore crucial to adapt the process to the specificity of each kind of SW solution (nature and  
718 concentrations of target pollutants, extracting agents and other compounds mobilized during  
719 the SW step).

720 Finally, the choice of the most suitable strategy for the treatment of a SW solution  
721 should be based on a cost-benefit analysis and life cycle analysis should be performed for  
722 selecting the most suitable strategy. Selective separation/degradation of target pollutants  
723 involve the implementation of costly processes (in terms of reagents and/or energy  
724 consumption). Therefore, these additional costs have to be compared to the savings coming  
725 from the reuse of the SW solution. The development of effective processes for recovery of  
726 extracting agents could also promote (i) the use of more expensive extracting agents with both  
727 higher extraction capacity and lower soil toxicity and (ii) the implementation of several  
728 successive SW steps in order to achieve higher removal rates of HOCs from real contaminated  
729 soils.

730

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736

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