

1 Article

2 **Electro-absorbers: A comparison on their** 3 **performance with jet-absorbers and absorption** 4 **columns**

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16 **Abstract:** This work focuses on the removal of perchloroethylene (PCE) from gaseous streams using
17 absorbers connected with electrolyzers. Two types of absorption devices (jet absorber and
18 absorption column) were compared. In addition, it has been evaluated the different by-products
19 generated when a simultaneous electrolysis with diamond anodes is carried out. PCE was not
20 mineralized but it was transformed into phosgene that mainly derivates into carbon tetrachloride.
21 Trichloroacetic acid was also formed but in much lower amounts. Results showed a more efficient
22 absorption of PCE in the packed column, which it is associated to the higher gas-liquid contact
23 surface. Jet absorber seems to favor the production of carbon tetrachloride in gaseous phase,
24 whereas the packed column promotes a higher concentration of trichloroacetic acid in liquid. It was
25 also evaluated the scale up of the electrolytic stage of these electro-absorption devices by using a
26 stack with 5 perforated electrode packages instead of a single cell. It has been tried to clarify the
27 effect of the applied current density on the speciation attained after the electrolysis of the absorbent.
28 Experiments reveal similar results in terms of PCE removal and a reduced generation of gaseous
29 intermediates at lower current densities.

30 **Keywords:** Perchloroethylene; absorption column; jet absorber; electro-absorption; scaleup

31

32 **Highlights**

- 33 - Significant differences in the speciation related to the absorption step.
- 34 - PCE was not mineralized but transformed into other products
- 35 - Phosgene and carbon tetrachloride are the main products with the jet absorber.
- 36 - Trichloroacetic acid is important when using the absorption column.
- 37 - Scaleup of electro-absorption with a stack cell was evaluated.

38 **1. Introduction**

39 In the recent years, many studies have been carried out to evaluate the removal of persistent
40 pollution from water and wastewater, but a reduced attention has been paid to the vapors emitted
41 during these treatments [1,2]. However, gaseous pollutants are considered an important problem for

42 the environment and for the human health [3,4] and their removal is really a matter of a major
43 importance. In this context, gaseous emissions associated to these streams in food waste treatments
44 plants have been studied over last decades, developing technologies capable to remove compounds
45 such as methyl mercaptan [5], hydrogen sulfide and trimethylamine [6,7], typically associated with
46 odor. Among them, it is important to highlight technologies such as thermal oxidation, selective
47 catalyst reduction, chemical scrubbing, bioscrubbing and biofiltration [8,9]. However, these
48 technologies have shown several disadvantages, such as catalyst deactivation and high operating
49 costs [10]. For this reason, other technologies such as scrubbing are gaining relevance, although it is
50 important to take in mind that this absorption process does not attain a complete removal but it only
51 promotes the transference of the gaseous pollutants into a liquid stream that needs for further
52 treatment [11]. In these systems the mass transfer is a key aspect and efficiency is controlled by
53 residence times and gas-liquid contact surfaces. So that, a design with good hydrodynamic behavior
54 is required using high and wide contactors [6,12-14].

55 Considering this necessity, recently [15], it was proposed the use of a jet absorber to promote the
56 gas-liquid contact in the treatment of a perchloroethylene stream. This system is based on the venturi
57 effect and avoid the use of a compressor, allowing an important reduction in the energy requirement
58 [16]. Obviously, this technique need to be combined with another destructive technology to remove
59 the pollutant from the liquid [17]. In this line, one of the options is the electrochemical technology in
60 which an electrochemical cell is combined with the absorption system in order to regenerate an
61 active catalyst in aqueous solution [18,19] or to produce an oxidant from salts such as phosphate [10]
62 or sulfate [20], which have been applied successfully to remove different gaseous pollutants.

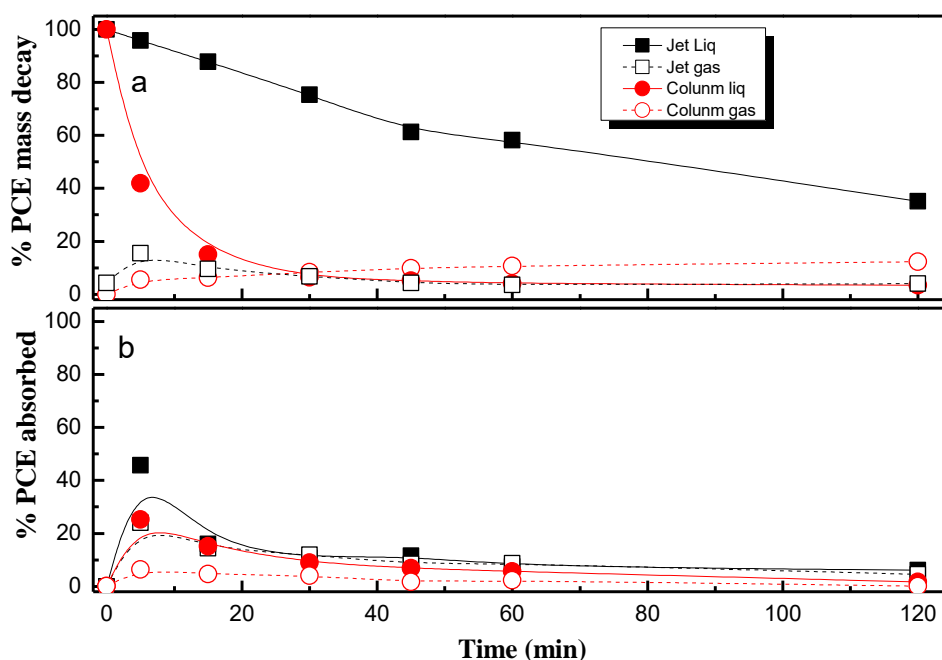
63 Because of their good physical properties as low flammability, chemical stability, and an
64 excellent washing solvent, PCE is frequently used in commercial processes such as machine
65 manufacturing, metal degreasing, and dry cleaning. As a result, many cases of PCE-contaminated
66 groundwater, soil [21] and indoor air of different stages of a wastewater treatment plant (WWTP) has
67 been reported. Considering its high toxicity, volatility, extremely persistence in the environment, as
68 well as the toxic reaction intermediates generate such as phosgene, chloroform and carbon
69 tetrachloride, PCE presents in water and/or air must be removed. Among the technologies developed
70 to eliminate PCE from liquids, it is worth to mention photocatalytic degradation [22], dielectric
71 barrier discharge plasma [23], phytoremediation [24], sonochemical degradation [25], electrochemical
72 degradation [26], sonoelectrochemical degradation [27,28], adsorption/electrolysis [29] etc. However,
73 these technologies are not valid for gaseous flows where, on contrary, the wet absorption process
74 could be considered as convenient and an economical technology. However, preliminary evaluation
75 of these coupled technologies with jet absorber showed a production of a vast number of
76 intermediates, mainly in the gaseous phase when PCE is used as a model compound with a negligible
77 concentration of carbon dioxide in comparison with intermediates as trichloroacetic acid or carbon
78 tetrachloride [15].

79 This work focuses on the comparison of the reactivity of gaseous PCE when it is absorbed using
80 a jet absorber and packed absorption column, coupled with an electrolyzer. In addition, two types of
81 electrolyzers were coupled, being one of them a single cell and the other a cell stack with five
82 electrodes. With this comparison it was tried to determine if the bottleneck of the scaleup is associated
83 to the efficiency of electrochemical devices.

84 2. Results and discussion

85 **Absorption using a packed column and jet absorber.** To determine the absorption capacity of
86 the two absorbers evaluated in this work, a single absorption process was carried out in the proposed
87 set-ups. To do this, PCE concentrations in liquid and gaseous phases were measured in both LWD
88 and AES tanks. Thus, in Figure 1, it can be seen the changes in the concentration of the PCE in the
89 tank where the simulated gaseous pollutant was produced (Part a) and in the tank that it is contained
90 the absorbent (Part b). As can be observed, the decay of PCE in the liquid phase of LWD tank depends
91 on the aeration and absorber used. Thus, in the system equipped with an air compressor and the
92 packed absorption column, PCE concentration decreases up to 95% whereas the system equipped

93 with the jet absorber is only capable to reduce the concentration around 35% after the same operation
 94 time (120 min). The effect of PCE condensation into the compressor was discarded in the results
 95 obtained with previous experiments taking some gaseous samples before and after this equipment
 96 for two hours obtaining no relevant differences in the mass balance of PCE. Thereby, the stripped
 97 PCE is transferred to the liquid circuit, where it is absorbed. The concentration of PCE absorbed
 98 reaches a maximum and, then, it starts to decrease. This seems to indicate that PCE is transformed
 99 into other products, whose relevance will be discussed afterwards. These results indicate that the
 100 absorption attained by the jet absorber is less efficient than that of the packed column and this
 101 observation can be explained considering two main factors, as a consequence of the higher gas-liquid
 102 surface area attained in the packed column because of the addition of solid spheres and the higher
 103 counter current gas flow promoted by the compressor installed (almost the double than in the jet
 104 absorber). In both systems, amounts of PCE in the gas phase are less important than in the liquid
 105 phase, but higher and fast absorption of PCE avoids the total degradation of this compound before
 106 the electrooxidation processes and this seems to point out an evolution of the system towards a
 107 different the gas-liquid equilibrium.



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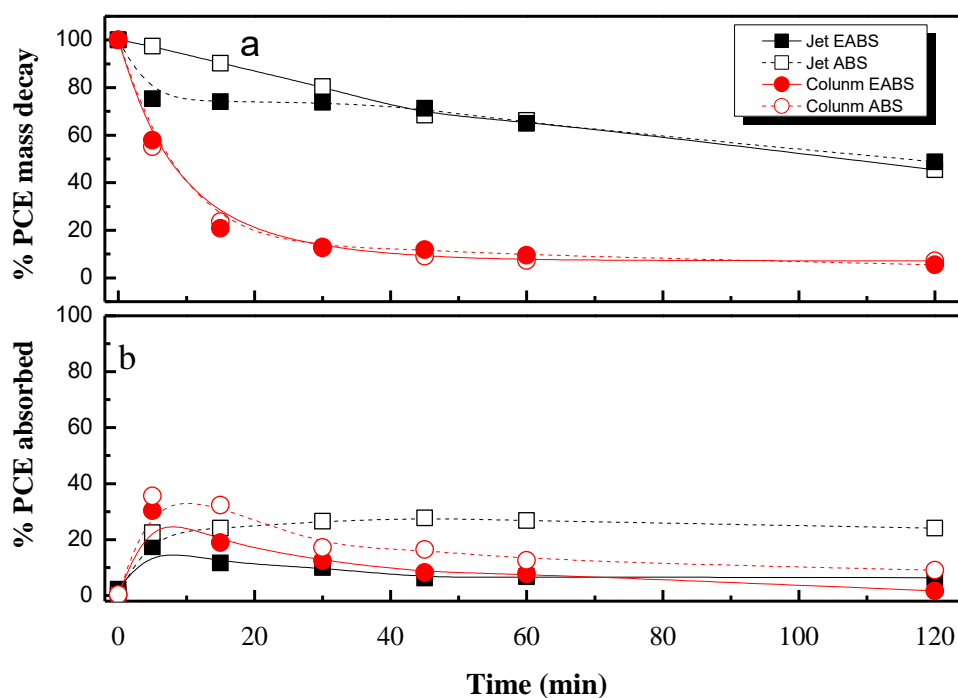
109 **Figure 1.** Changes in the PCE mass during the absorption using the jet absorber (■, □) and the
 110 absorption packed column (●, ○) in a) LWD tank containing liquid waste polluted with PCE and b)
 111 AES tank.

112 **Performance of electro-absorbers.** Once it was determined the higher efficiency of the packed
 113 column in terms of absorbing the PCE released from the wastewater, it was necessary to determine
 114 the viability of the electrochemical process to modify the composition of the pollutants absorbed.
 115 Thus, in Figure 2, it is shown the changes in the removal of PCE (in terms of aggregated values of
 116 liquid and gaseous phases) in both tanks of the two systems, during absorption (ABS) or electro-
 117 absorption (EABS) tests.

118 As it can be seen, in the liquid waste desorption tank (LWD tank), over the complete test in the
 119 system equipped with the packed column absorber no differences are observed with the application
 120 of current to the electrochemical cells. On contrary, minor differences appear in the removal of PCE
 121 when jet absorber was used. During the electrochemical process, a faster depletion of PCE was
 122 observed during the first 30 minutes. However, after this initial change, the response is stabilized in
 123 similar values of those attained by the non-electrochemical absorption process.

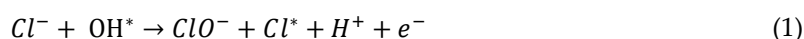
124 In the tank containing the absorbent (AES tank), there are important differences between both
 125 absorption systems. In the case of the jet absorber, evolution in the concentration of PCE is totally

126 different, although it increases fast at the beginning in both systems, in the single absorption was
 127 obtained almost 30% of PCE recovery at the end, and in the electroabsorption process, the evolution
 128 of the PCE decreased to detect around a 5% of PCE volatilized. This behavior would be influenced
 129 by the reactivity promoted by the electrochemical process during the absorption. Regarding to the
 130 process carried out with packed column absorber, the same trends are observed in both cases, with
 131 fast absorption of PCE at the early values, but less than 15 % of PCE absorbed at the end. Results
 132 confirm that the liquid-gas contact is more efficient in the column absorber and indicate that the effect
 133 of the electrochemical reactions in the global process are not as important as in the case of jet absorber.



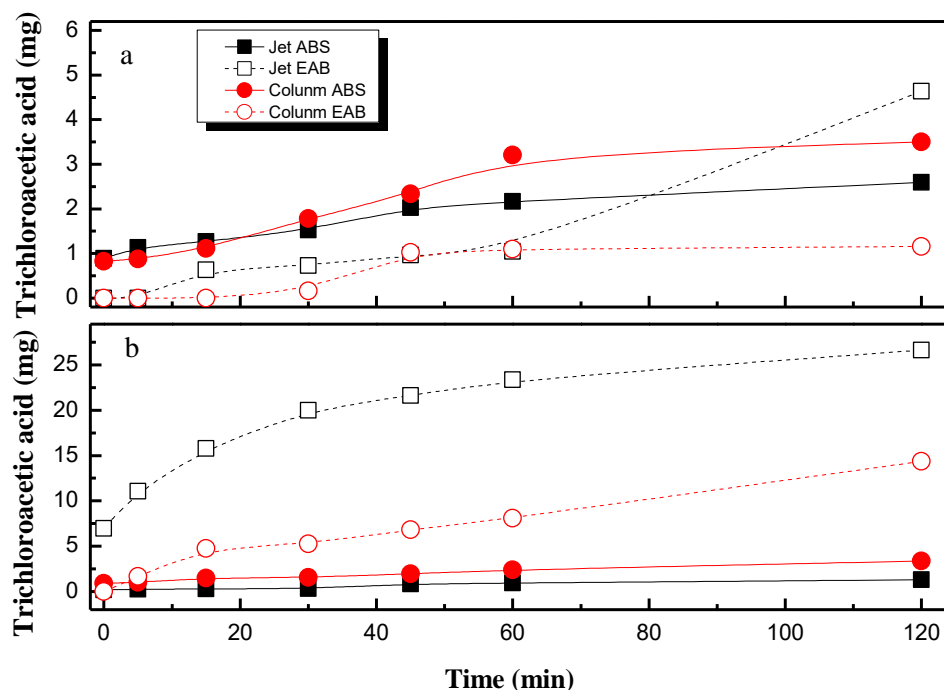
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 135 **Figure 2.** Evolution of PCE as a function of the time in the jet absorber system (■) and in the packed
 136 column (●) during the absorption (ABS) and the electro-absorption process (EABS). a) LWD tank. b)
 137 AES tank.

138 During the absorption processes, several by-products were detected by HPLC, related with the
 139 removal pathway followed in aqueous solutions. One of them is the trichloroacetic acid (Figure 3)
 140 that corresponds to an acidic compound derived of the single displacement of a chloride radical from
 141 the PCE and the hydroxyl radicals obtained in the electrode surface [33,34], as it is shown in the eqs
 142 (1-4).



143 This intermediate was detected in both tanks which means that just the instability of PCE in
 144 water promote this compound. However total amount detected in LWD tank is lower than 5 mg with
 145 soft increasing trends with the time. In the absorbent tank, higher concentration is detected during
 146 both electro-absorption processes, which means that the secondary products generated in the electro-
 147 oxidation could promote also an hydrogenolysis that increase the total concentration of this
 148 intermediate. Trichloroacetic acid due to its high polarity and low vapor pressure, is commonly found
 149 in liquid phase. Thereby, the higher generation also in the system equipped with the jet absorber

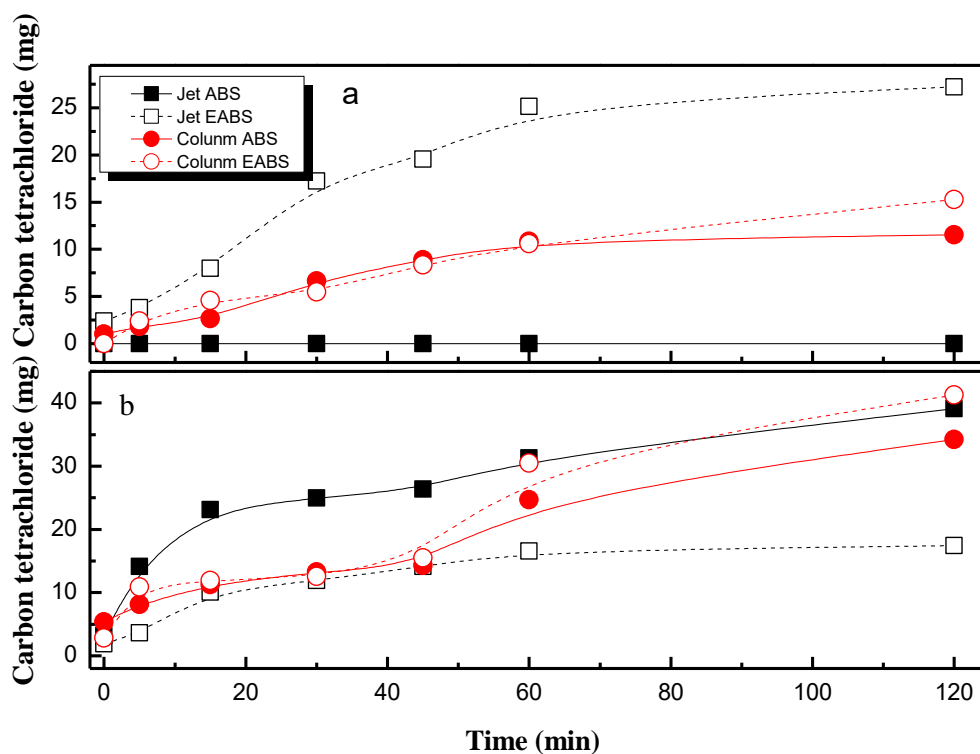
150 suggests that an important transformation of PCE is transferred directly to the liquid phase which
 151 makes it easier the further pollution removal [35].



152

153 **Figure 3.** Evolution of trichloroacetic acid as a function of the time in the jet absorber system (■) and
 154 in the packed column (●) during the absorption (full points) and the electro-absorption process
 155 (empty points). a) LWD tank. b) AES tank.

156 Another intermediate detected by HPLC analysis is the carbon tetrachloride, and its behavior is
 157 shown in **Figure 4**. This compound shows similar concentrations in the waste and the absorber tanks,
 158 being undetectable in the waste tank (LWD) when the jet absorber system without electric current
 159 was used. Its formation can be explained in terms of the decomposition of phosgene, which is an
 160 intermediated known to be produced by wet hydrolysis of PCE [36]. This compound behaves as a
 161 final product because no decreasing trends were obtained with the time and it was produced without
 162 remarked differences in both ABS and EAB processes.



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165 **Figure 4.** Evolution of carbon tetrachloride as a function of the time in the jet absorber system (■) and
 166 in the packed column (●) during the absorption (full points) and the electro-absorption process
 (empty points). a) LWD tank. b) AES Tank.

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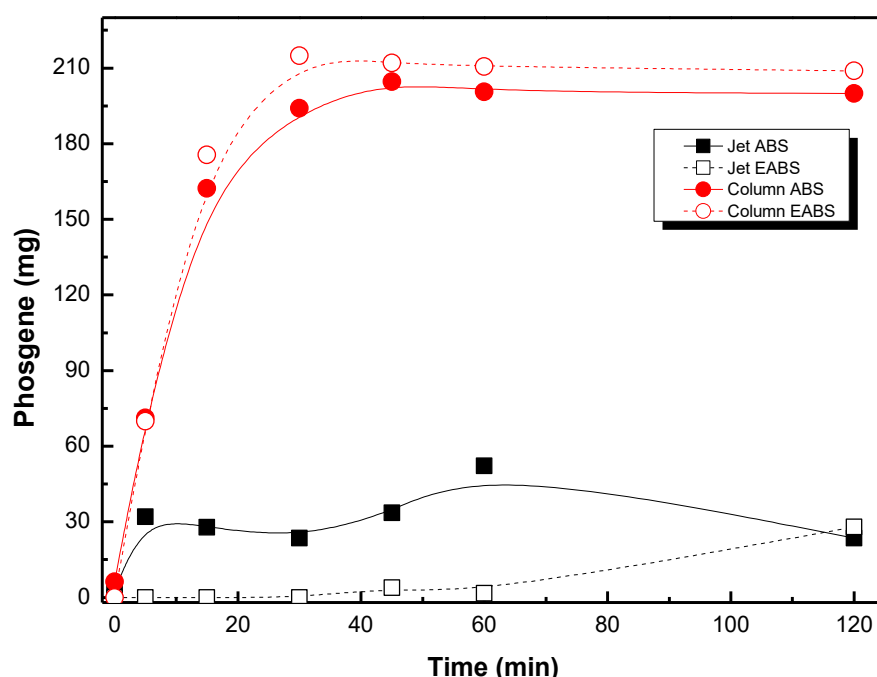
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Both intermediates, carbon tetrachloride and trichloroacetic acid were the unique compounds detected and quantified by HPLC, but they represent different **removal pathways** of PCE related to the higher gas-liquid contact or to the promoted attack of strong oxidants **derived from generated during** the electro-oxidation. However, mass balance informs about the existence of a third compound with absorption at 365 nm [37] and with a ratio chlorine / carbon 2:1, which is not detected neither by GC nor HPLC. This information is **consistent compatible** with the formation of phosgene, an organic compound **formed produced** by wet hydrolysis of PCE [38],[36]. **Figure 5** shows **of** the amount of phosgene estimated by carbon-mass balance. As can be observed, there is a clear difference in the evolution of phosgene in tests carried out with the jet absorber and the packed column. In the electro-absorption with jet aerator, the concentration of phosgene is negligible which agrees with the higher efficiency showed in the PCE recovery and the use of electro-oxidation with diamond electrodes. Regarding to the Jet ABS, although the evolution of phosgene seems higher than the process applying current density, at the end, similar concentration was reached which shows that phosgene generation is not very influenced by the presence of the electrolytic processes. On the other hand, the evolution of phosgene was similar in the Column ABS and Column EABS processes, but it was very different in comparison with Jet ABS and Jet EABS, reaching a total mass around 5 times higher. This behavior is associated to the different absorption setups used and the different pathways that control **s** the depletion of PCE in each absorption process.



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186 **Figure 5.** Evolution of phosgene as a function of the time in the jet aerator system (■) and in the
 187 packed column (●) during the absorption (full points) and the electro-absorption process (empty
 188 points).

189 Once it was monitored the main products obtained, different **removal** pathways of the
 190 degradation of PCE could be proposed. The distribution of these compounds detected at the end of
 191 the process are shown in Table 1. It is highlighted that only carbon dioxide was detected after the
 192 absorption with **the jet absorber (Jet ABS)** but also in this set up was obtained the lower removal of
 193 PCE. The most effective system for the removal of PCE was the packed column (7.89% - 9.21%
 194 respectively). However, in the case of jet absorber, it is shown a high concentration of trichloroacetic
 195 acid in Jet **EABS** test. In recent studies [23,39] it was reported the mechanism of oxidation of PCE to
 196 trichloroacetic acid, as well as the formation chlorine radicals that can attack to the carbon cleavage
 197 of the PCE to form phosgene. This compound is unstable and could be degraded into carbon
 198 tetrachloride and carbon dioxide.

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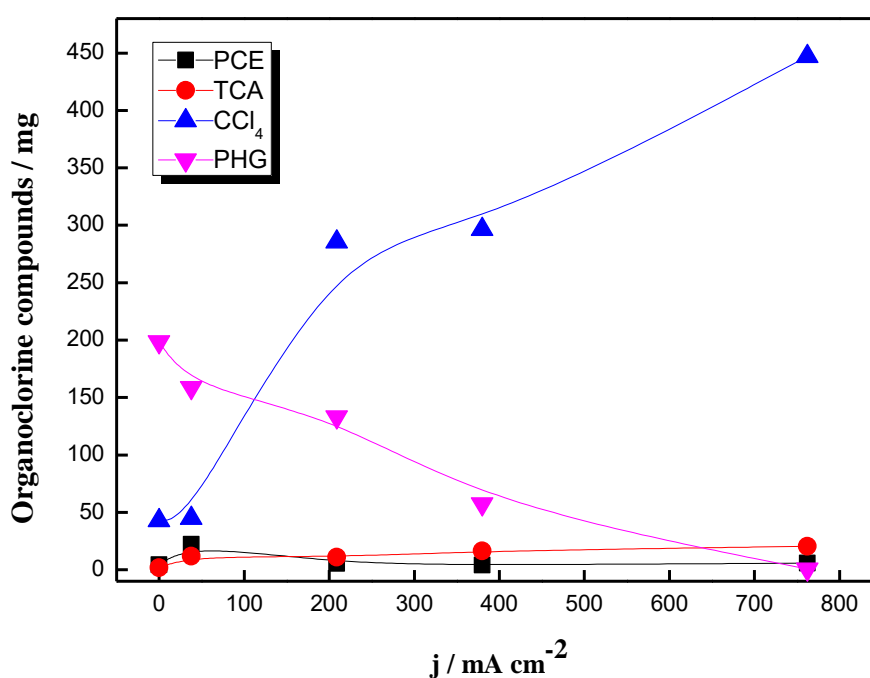
Table 1. Distribution of intermediates in carbon conversion percentage at the end of the treatment with the different technologies proposed.

Degradation compounds	[1]	[2]	[3]	[4]
carbon tetrachloride	11.16%	14.45%	11.41%	12.49%
trichloroacetic acid	2.79%	20.34%	3.22%	6.46%
phosgene	9.15%	16.07%	77.47%	71.83%
perchloroethylene	59.78%	49.14%	7.89%	9.21%
carbon dioxide	17.12%	0.00%	0.00%	0.00%

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202 [1] Absorption with jet absorber (Jet ABS), [2] Electro-absorption with jet absorber (Jet EABS), [3]
 203 Absorption with packed column (Column ABS), [4] Electro-absorption with packed column (Column
 204 EABS).

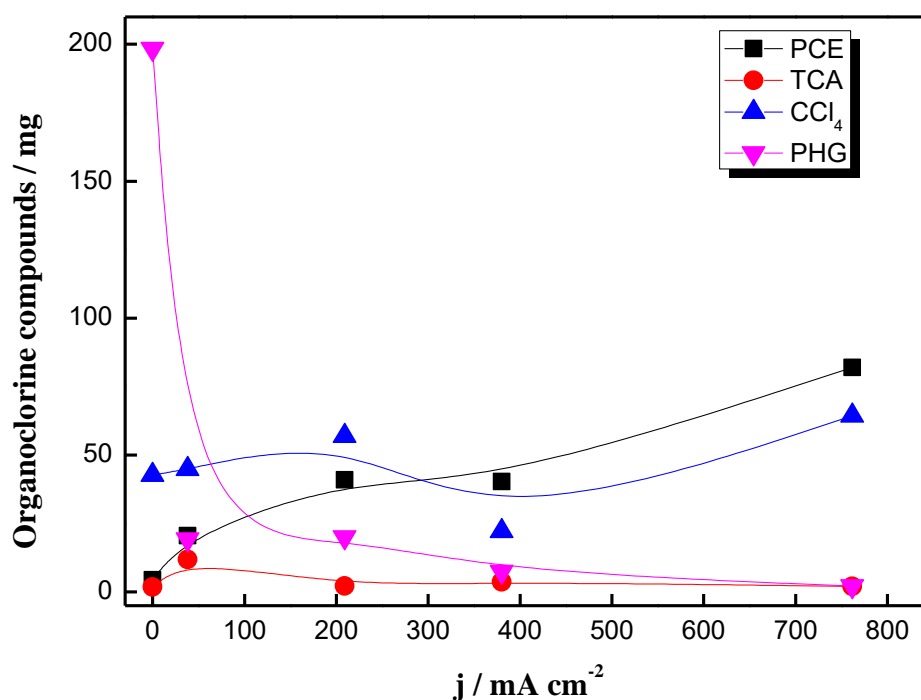
205 **Scaleup of electro-absorption processes.** To evaluate the scaleup of the electrolytic process, the
 206 system equipped with the packed column absorber was selected for an additional study, due to its
 207 better performance. In this new study, the single cell was replaced by a stack of cells equipped with
 208 five flow through Diachem® anodes and ten stainless steel cathodes (Condiacell®). Figure 6 shows
 209 the total mass of PCE (sum of liquid and vapor phase) and by-products generated during the electro-
 210 absorption process, as a function of current density applied after a treatment of 2 hours. The total
 211 mass of PCE does not vary with the current density used in the electrochemical process. In fact, it is
 212 nearly depleted. Surprisingly, TCA is only observed at low concentrations, which may indicate a
 213 different performance of the stack with this product as compared with the single electrolyzer. The
 214 main products are phosgene and CCl₄. However, substantial modifications were observed in their
 215 distribution. As can be seen, at low current densities, the main final reaction product is phosgene,
 216 remaining in solution around 200 mg after the treatment. When the current density increases, CCl₄
 217 becomes the primary final product (up to 450 mg), which it is probably motivated by the side
 218 reactions that occurs in the electrode surface due to the limitations of mass transfer. Nevertheless, in
 219 this point it is important to point out that this figure compares results which correspond to different
 220 electric charged passed, because the electrolysis times in all experiments were the same. Thus, the
 221 higher is the current density applied, the higher is the electric charge supplied in each test and, hence,
 222 the progress of the electrochemical reaction. So, despite having used the same treatment times, results
 223 may be indicating that phosgene is produced previously to carbon tetrachloride and not the real effect
 224 of the current density.



225
 226 **Figure 6.** Organochlorinated compounds generated at the end of treatment during the electro-
 227 absorption technology using a Condiacell® as function of current density applied (■) PCE, (●) TCA,
 228 (▲) CCl₄ and (▼) phosgene (PHG).

229 To clearly see this effect, it is necessary to compare results obtained at the same electric charge
 230 passed. This can be seen in Figure 7, in which all tests were compared for an applied electric charge
 231 of 0.35 Ah dm⁻³ and where it can be seen that higher current densities are less efficient in the
 232 degradation of PCE, which can be explained in terms of the promotion of side reactions such as water
 233 oxidation and reduction. Meanwhile, low current densities promote the formation of phosgene.

234 Amounts of CCl_4 produced do not depend on the current density, being the key product in all tests.
 235 Relevance of TCA is very low and limited to the application of low current densities.



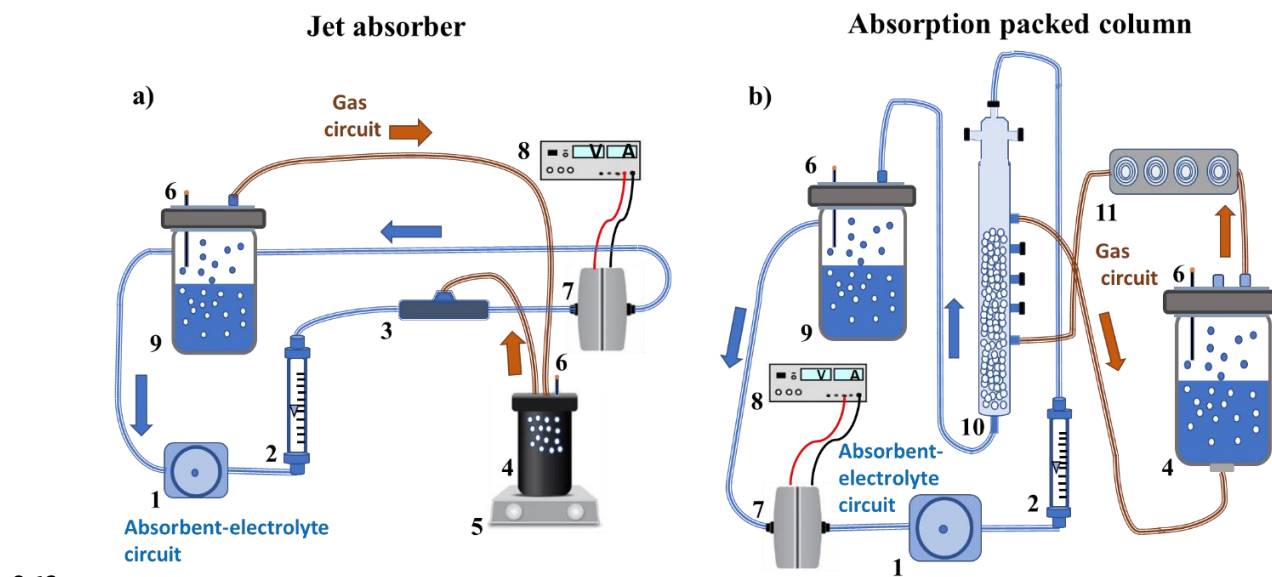
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 237 **Figure 7.** Influence of the current density on the products generated at 0.35 Ah dm^{-3} during the electro-
 238 absorption technology using a Condiacell®. (■) PCE, (●) TCA, (▲) CCl_4 and (▼) phosgene (PHG).

239 In this way, this study indicates that the electro-absorption using a cell stack could be a feasible
 240 technology to achieve good PCE oxidation. However, further research should be developed due to
 241 no complete elimination of its by-products was attained. No phosgene was found at the end of the
 242 process, although significant concentrations of carbon tetrachloride remain in solution mainly when
 243 higher current densities are applied. Thereby, it is important to warn about the toxicity of phosgene
 244 and the reduction of their concentration which is a considerable achievement of the electrochemical
 245 technology. However, the removal of carbon tetrachloride that is a recalcitrant by-product would be
 246 in subsequent studies the real challenge to achieve a complete mineralization.

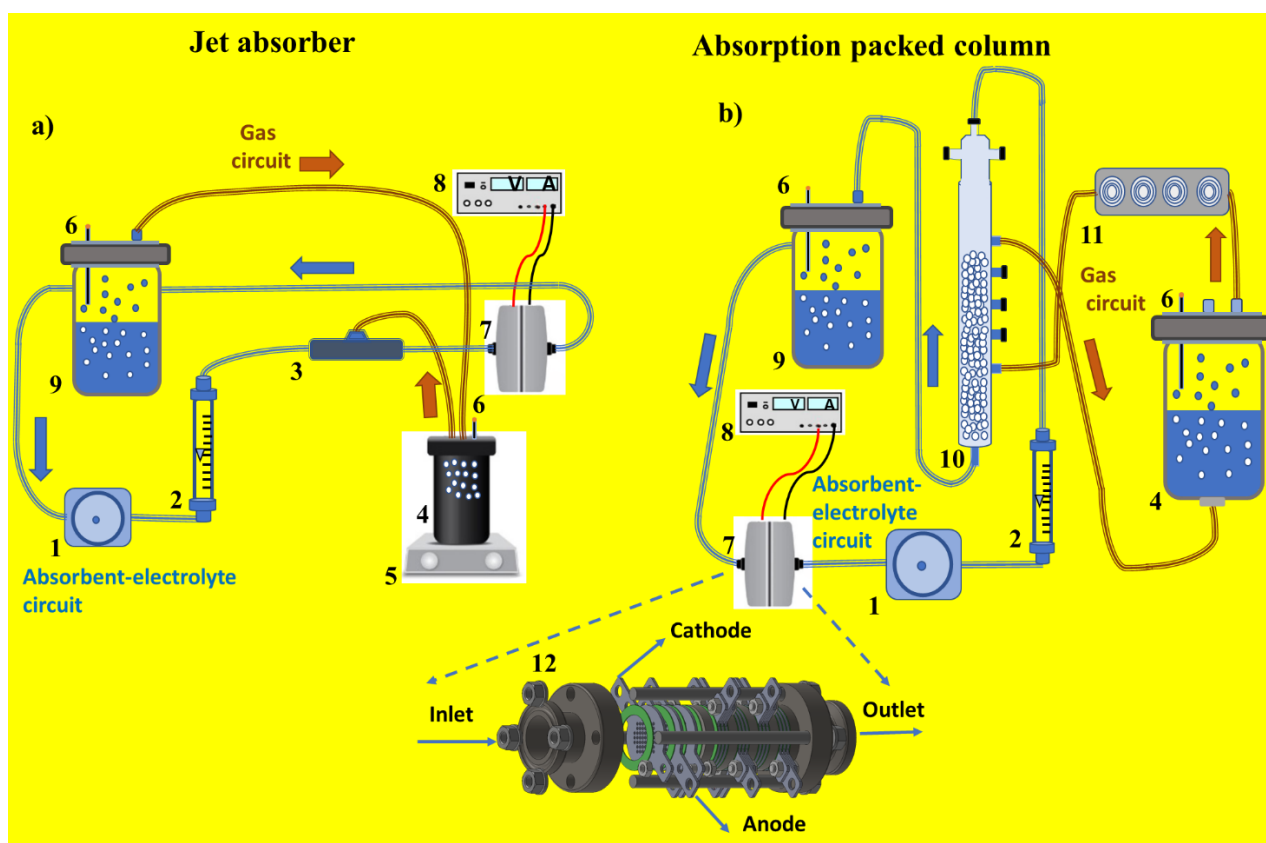
247 3. Materials and methods

248 **Chemicals.** All chemical reagents, including perchloroethylene ($\text{C}_2\text{Cl}_4 > 99\%$) (a.r., Sigma-
 249 Aldrich), anhydrous sodium sulphate, phosphoric acid (85%), sulfuric acid (99.8%), sodium
 250 carbonate (a.r., Fluka, Spain), were analytical grade and used as received. Hexane HPLC grade (a.r.,
 251 Sigma-Aldrich, Spain) was used as mobile phase in gas chromatography. Helium and nitrogen (Al
 252 Air Liquide España, S.A) were used in gas chromatography and helium was filtered by a
 253 Hydrocarbon Cartridge Filter (Thermo Fisher Scientific) before its use. Double deionized water
 254 (Millipore Milli-Q system, resistivity = $18.2 \text{ M}\Omega \text{ cm}$ at $25 \text{ }^\circ\text{C}$) was used to prepare all solutions.

255 **Experimental setup.** Absorption and electro-absorption processes were developed in the two
 256 versatile set-ups shown in **Figure 8**: one in that integrates a jet absorber (part a) and another that
 257 integrates an absorption packed column (part b). Both experimental systems are divided into two
 258 connected circuits: the liquid (absorbent-electrolyte) circuit and the gas circuit. In both cases there
 259 is a tank (liquid waste desorption (LWD) tank, (4) where the raw concentrated solution (1 L of
 260 aqueous solution with 150 mg dm^{-3} of perchloroethylene (PCE)) is stored. From here, the
 261 volatilization and stripping of PCE is induced by a gas flow.
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Figure 8. Schematic diagram for the absorption and electro-absorption process using both setups evaluated: a) jet-absorber. b) absorption packed column. 1. Pump 2. Rotameter 3. Jet-aerator 4. Liquid Waste desorption (LWD) tank 5. Magnetic stirrer 6. Sampling point 7. Electrochemical cell 8. Power supply 9. Absorbent electrolyte storage (ABS) tank 10. Glass packed column 11. Air-compressor silent pump. 12. Stack of cells model ECWP d20X5P.

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The jet absorber is based on the venturi effect and it is used to create suction favoring the flow of gas from tank 4 into the liquid circuit. So, the stripping of the PCE from the LWD tank (4) is promoted. The small throat diameter of the jet (4.23 mm) modifies the size of the bubbles generated, affecting to the behavior of the absorption process. Additionally, to ensure good mixing conditions in tank 4, a magnetic stirrer (5) was used [15]. In the other system (Figure 1b), the gas phase is

275 generated with an air-compressor (Silent Pump (11) model SI6000, ICA SA Spain) with 3.8W and a
276 flowrate of 360 L h⁻¹. This gas stream passes through the LWD tank (4), favoring the stripping of PCE
277 and then it flows through the absorption packed column (10) where it is transferred to liquid circuit.
278 Gas and liquid phases flow countercurrent in the column. The absorption column is made of glass
279 with 0.5m long and an inner diameter of 500 mm. A length of 0.4m of the column was packed with
280 glass spheres (8 mm ± 0.75). These spheres are solid (2.5g cm⁻³) made of high-quality borosilicate glass
281 which are a good choice for demanding corrosive environments. Therefore, the role of tank 4 in both
282 cases is to produce a more realistic gaseous pollutant flow, with real gaseous mixtures water-PCE,
283 which can influence on the later treatment because of the reactivity of PCE in wet environments [30].

284 On the other hand, the liquid phase (electrolyte solution with the PCE absorbed) is pumped with
285 a centrifugal pump (1) to the electrochemical cell (7), that is connected to a power supply (8) and
286 where the degradation of the species present in the liquid phased occurs. Additionally, in this system
287 conventional electrochemical cell is modified to test the scaleup of the process using a stack of cells
288 (12) model ECWP d20X5P. The absorbent-electrolyte storage (AES) tank (9) is the auxiliary tank of
289 the electrochemical cell in both setups. Its function is to provide residence time to the electrochemical
290 cell in order to promote chemical reactions mediated by oxidants or reductants generated by the
291 application of the electric current. In both systems, there is a connection between the AES and LWD
292 tanks (9 and 4) to equilibrate the total pressure of the system.

293 Both AES tank (6) and LWD tank (4) were made of polyvinyl chloride (PVC) with capacities of
294 2.5 L and 1.2 L, respectively. Sample points are implemented in both tanks. The liquid phase used as
295 absorbent was an aqueous solution of Na₂SO₄ (0.1 mol L⁻¹). The centrifugal pump is a Micropump®
296 GB-P25 J F5 S (flow rate 160 L h⁻¹) connected to the electrochemical cell by a Tecalan® tube. Extreme
297 care was taken to avoid gaseous losses in all compartments by using tight-fitting ground silicone
298 stoppers and by sealing with Teflon tape.

299 To study and compare the two absorption systems proposed in terms of absorption and
300 depletion of PCE, experiments were performed for 120 minutes collecting samples in duplicated at
301 specified time intervals.

302 Electrochemical processes were conducted with two kind of commercial electrochemical cells.
303 The first one was a DiaCell® supplied by Adamant Technologies (Switzerland) which conductive-
304 diamond electrodes (p-Si-boron-doped diamond) were used as anode and cathode, respectively.
305 Both electrodes were circular (100 mm diameter) with a geometric area of 78.6 cm². The BDD coating
306 have a 2-3 μm of the thickness, boron concentration 500 ppm, and the relation sp³/sp² >150. The active
307 surface is 78.6 cm² and the inter electrode gap is 1 mm. The stack of cells selected for the scaleup
308 was a CondiaCell® model ECWP d20X5P supplied by Condias GmbH (Germany) consisting of ten
309 circular stainless-steel electrodes as cathodes with 20x1.5 mm and five circular Diachem® type
310 perforated electrodes as anode. These diamond electrodes (50 × 24 × 1.3 mm³) were built on a
311 substrate that consists of a niobium mesh (type B) and were assembled in two stacks with a
312 NAFION® cation exchange membrane separating the anode and cathode and acting as the electrolyte
313 [31]. The anodic active area per package is approximately 420 mm² and the inter electrodes gap is 0.5
314 mm. Four current were studied being 0.8, 4.4, 8 and 16 A. Both kind of electrodes were subjected to
315 cleaning procedure during 10 min in a 1 M Na₂SO₄ solution at 15 mA/cm² prior to electrolysis assays.
316 All the processes were conducted at room temperature (20 ± 2 °C) and at atmospheric pressure
317 conditions.

318 **Analysis procedures.** Liquid and gas samples were taken from two different sampling ports
319 which are placed in the accumulation and PCE tanks. To determine and quantify PCE concentration
320 in gas and liquid phase, it has been followed the procedure described elsewhere [15,32].

321 For the determination of by-products produced in the absorption and electro-absorption
322 treatment, two chromatographic methods were employed. The first one, was used to analyze carbon
323 tetrachloride by means Jasco HPLC LC-2000 with a PDA MD-2018 Detector (Jasco, Japan). The mobile
324 phase consisted of an aqueous solution of 0.1% of phosphoric acid (flow rate of 1.0 mL min⁻¹). The
325 detection wavelength used was 280 nm and the temperature of the oven was maintained at 25 °C.
326 Volume injection was set to 20 μL. The second method was used to analyze trichloroacetic acid, which

327 was determined using a HPLC Agilent 1100 series (Agilent Tech. USA) with a detection wavelength
328 of 220 nm. The ion exchange column used was Supercogel™ H Column with 30cm x 7.8mm ID. The
329 mobile phase: 1% phosphoric acid (H₃PO₄), column temperature: 30°C, flow rate: 0.8 mL/min;
330 injection volume: 20 µL.

331 4. Conclusions

332 From this work, the following conclusions can be drawn:

- 333 • Both, jet absorbers and packed column absorbers, can be used for the absorption stage of the
334 removal of PCE with electro-absorbers. Packed columns reached more successful results.
- 335 • The column absorber favors the formation of trichloroacetic acid.
- 336 • Electro-oxidation process with diamond electrodes increases the reduction of PCE but
337 enhances the generation of more dangerous and toxic intermediates. Trichloroacetic acid and
338 carbon tetrachloride are the main compounds detected. A removal pathway for PCE
339 degradation related to the absorption efficiency in both set-ups can be proposed.
- 340 • The use of a cell stack with five electrodes do not show remarkable differences in the removal
341 efficiency of PCE as compared to the singles cell. However, TCA formation is not promoted
342 with the scaled-up system. In addition, current density affects importantly to results. Low
343 current densities lead to the formation of higher amounts of phosgene and higher current
344 densities to the less efficient removal of PCE.

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