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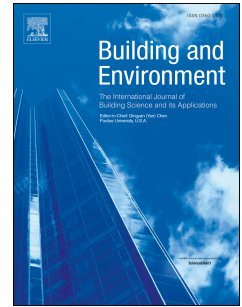
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1 Fast Sorption Measurements of VOCs on Building 2 Materials: Part 2 – Comparison between FLEC and 3 CLIMPAQ Methods

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19 **Abstract**

20 A new method was developed to measure on the field VOC sorption coefficients (k_a ; k_d) on
21 the surface of a material by coupling a Field and Laboratory Emission Cell (FLEC) to a
22 Proton Transfer Reaction-Mass Spectrometer (PTR-MS) as presented in the first part of this
23 study. In this second part, the method is compared to the classical method based on a
24 CLIMPAQ chamber coupled to an on-line GC analyzer. Different models were used to
25 determine the sorption parameters from experimental data taking into account the sink effect
26 on empty chamber walls and the presence of a boundary-layer. Determined sorption
27 equilibrium coefficients K_e (k_a/k_d) for a mixture of BTEX on a gypsum board was found to be
28 in good agreement between both methods. However, the CLIMPAQ method seems to be less
29 robust than the FLEC method in the determination of sorption coefficients since more than
30 one couple of (k_a ; k_d), showing the same ratio K_e can retrieve the same CLIMPAQ
31 experimental data. Giving this result, the question arises about the reliability of the literature
32 data determined using emission test chamber which could be one of the reasons behind the
33 discrepancies found between experimental indoor concentrations and predicted ones using
34 chamber derived parameters.

35 **Key words**

36 *CLIMPAQ, FLEC, Sorption, Building materials, Indoor Air Quality, model*

37 1. Introduction

38 Nowadays, people spend between 60-80% of their time in indoor areas that can be
39 contaminated by a large range of pollutants having hazardous effects on human health [1-4].
40 Pollutants, especially Volatile Organic Compounds (VOCs), are ubiquitous contaminants for
41 indoor areas [5]. Other than their emissions by surfaces such as building materials, VOCs can
42 interact with these indoor surfaces through different processes including adsorption and
43 desorption. Therefore sorption processes can be key drivers of indoor air concentrations since
44 building materials can act as both a source or a sink for VOCs [6].

45 In the literature, studies were carried out using either static or dynamic (flow-through)
46 chamber experiments to determine sorption rate coefficients (k_a and k_d) of VOCs on indoor
47 materials or equilibrium partition coefficients (K_e) [7-10]. In these studies, experimental data
48 were analysed using different mathematical sinks models to extract sorption parameters [11,
49 12]. However, indoor air quality models using chamber derived parameters failed to predict
50 real indoor concentrations [13] and differences as high as a factor of 9 were observed [14].
51 First, this disagreement can be related to the non-representativeness of laboratory experiments
52 compared to field conditions (type of material, implementation conditions, aging due to
53 environmental conditions, etc.) and highlights a need to reliably measure sorption parameters
54 in the field under real conditions [14]. Secondly, sorption on experimental chamber internal
55 walls was considered in some studies as insignificant [15] while others [7, 16-18] reported a
56 sink effect on chamber walls for different VOCs such as ethylbenzene, n-dodecane, α -pinene,
57 1,2,4-trichlorobenzene...etc. Thus, any underestimation of the chamber sink can introduce
58 biases in measuring sorption coefficients of materials. Thirdly, the use of inappropriate
59 mathematical models to extract sorption coefficients from experimental data may also
60 introduce biases that could be responsible for the discrepancies mentioned above. Blondeau
61 [11] demonstrated that models relating macroscopically the bulk air and the surface
62 concentrations through adsorption and desorption constants are not scientifically sound.
63 Zhang YP [19] highlighted that the mass transport process taking place between the material
64 surface and the bulk air has to be accounted for, which is not usually described in the
65 mathematical models used to analyse chamber experiments. In fact, several models has been
66 developed without taking into account for the mass transfer coefficient but the adsorption (k_a)
67 and desorption (k_d) rates [9, 10, 20] or the diffusion coefficient (D_m) in the building material
68 [21]. Later on, Deng [22] proposed an improved model that considers for the convective mass
69 transfer coefficient (h_m) through the boundary layer present on the surface of a material as
70 well as the diffusion and the partitioning coefficients. Therefore, models combining local
71 sorption equilibriums should be used in combination with the mass transport equations to fit
72 the experimental measurements.

73 Trying to shed some light on the discrepancies found in literature, a new methodology based
74 on a coupling between a FLEC (Field and Laboratory Emission Cell) and a PTR-MS (Proton
75 Transfer Reaction – Mass Spectrometer), was developed. The experimental setup is detailed
76 in the companion paper (Part 1) [23], and only few details are given here. The FLEC inlet is
77 connected to two gas generation systems, to be supplied either with humid clean air at
78 constant flow rate and stable relative humidity or with a diluted VOC mixture; the outlet is

79 connected to the PTR-MS to quantify VOC concentrations exiting the cell. A sorption
80 experiment performed using the FLEC is similar to the classical experiments described in the
81 literature and using a test emission chamber of several liters of volume. The method was
82 tested by performing sorption experiments on a gypsum board and vinyl flooring using a
83 mixture of BTEX at ppb levels. Adsorption and desorption coefficients were derived from
84 experimental output concentration profiles using the Tichenor model [10] that can be applied
85 to the FLEC cavity to determine elementary sorption coefficients. Sorption coefficients were
86 successfully determined and independently on flow conditions and on VOCs concentration,
87 with an experimental error lower than 15%. The limits of applicability were also assessed for
88 this method and showed that sorption parameters (k_a , k_d) in the range of ($0.01\text{m}\cdot\text{h}^{-1}$; 0.01h^{-1})
89 and ($0.09\text{m}\cdot\text{h}^{-1}$; 0.09h^{-1}) can be measured using 2 and 20 seconds of time resolution
90 respectively, with an accuracy better than 10%. The FLEC-PTRMS method is suitable for
91 field applications and allows a reduction of the measurement time to 0.5-12 hours compared
92 to several days for the emission test chamber method [7, 24, 25].

93 The objective of the present study is to compare sorption coefficients measured using the
94 FLEC-PTRMS and the emission test chamber methodologies for a common VOC mixture and
95 the same unpainted gypsum board. In fact, the FLEC was developed and used in previous
96 studies to measure materials emissions [26] as an alternative method to emission test
97 chambers [27]. Under this framework, the two methods were only compared for emission
98 measurements in several studies [28-30]. Since the FLEC had never been used as a tool to
99 measure *in-situ* VOC sorption parameters, a comparison for sorption measurements has yet to
100 be performed. To achieve this new objective, a 50-liter CLIMPAQ chamber (Chamber for
101 Laboratory Investigations of Materials, Pollution and Air Quality) coupled to a compact gas
102 chromatography analyser (*AirmoVOC*, *Chromatotech*) was used to perform the sorption
103 experiments. The comparison of the two methods involved two approaches:

- 104 (1) the comparison between CLIMPAQ experimental concentration profiles and the
105 concentration profiles that should be obtained considering the sorption parameters derived
106 using the FLEC-PTRMS method and,
- 107 (2) the comparison of the sorption coefficients derived from the CLIMPAQ data using
108 two models (that account for the sink effect on the chamber walls and for the boundary-layer
109 mass transport) with the FLEC derived parameters.

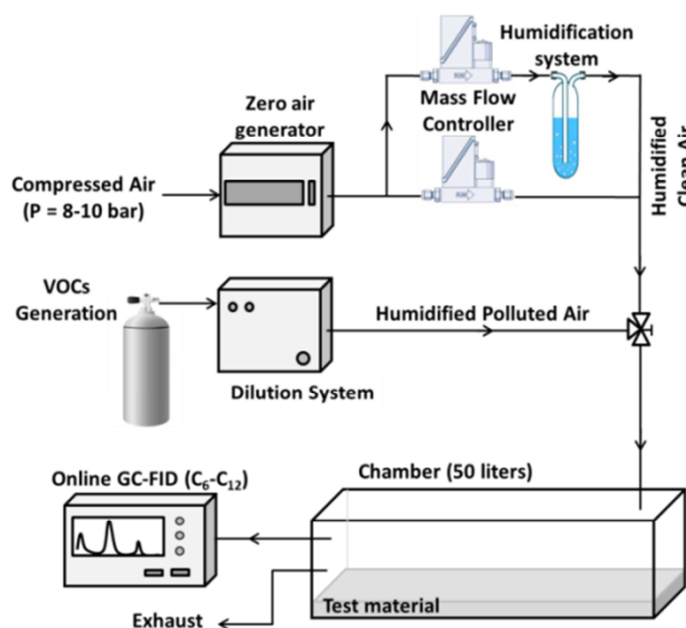
110 A highlight on the usefulness of the FLEC derived parameters for modelling sorption
111 processes in real buildings is also given.

112 **2. Materials and Methods**

113 To perform the comparison experiments between FLEC and CLIMPAQ methodologies, both
114 experiments are conducted using the same BTEX mixture provided by Air products and
115 containing the following VOC in the mixing ratio of 2 for benzene, 6 for toluene, 1 for ethyl
116 benzene, 2 for p-xylene and 1 for o-xylene. The same test material is used, a 12.5 mm thick
117 unpainted gypsum board. In the part 1[23], the use of two samples coming from the same
118 material showed no significant difference on measured sorption parameters. Since the FLEC
119 experiment was described in the first part of this study [23], only the CLIMPAQ experiments
120 are presented in this section.

121 **2.1. Experimental setup**

122 The experimental setup used in this part of the study is based on the coupling of a CLIMPAQ
 123 chamber to an on-line GC analyser and presented in Figure 1.



124

125 **Figure 1. Experimental setup for measuring VOCs sorption coefficients on building materials,**
 126 **using a CLIMPAQ test emission chamber.**

127 The CLIMPAQ chamber [27] is made of panes of 6 mm window glass assembled with low-
 128 emitting epoxy glue and has a volume of 50.9 litres. Other main surface materials used are
 129 stainless steel and aluminium. One internal fan recirculates air over the test material. The
 130 chamber outlet is connected to on-line gas chromatograph (GC) equipped with a flame
 131 ionization detector (FID) (*Airmo VOC C6C12. Chromatotec*). Air samples are collected every
 132 30 min at a flow rate of 60 mL/min for 2 min. The sampling flow rate is controlled by a
 133 critical orifice of 100 μm diameter. Hydrocarbons are preconcentrated at ambient temperature
 134 on a glass trap filled with CarbotrapB. After sampling, the trap is heated to 190°C in order to
 135 desorb and inject the VOCs into an analytic column MXT30CE (30 m \times 0.28 mm \times 0.1 μm)
 136 connected to the FID. The calibration coefficients are determined using a certified standard
 137 mixture provided by NPL (*National Physical Laboratory, UK*) and are used to convert the
 138 peak areas into concentrations in $\mu\text{g}/\text{m}^3$. The measurement uncertainty is not evaluated in
 139 this work but was determined in a previous study and is approximately 15% [31]. Using the
 140 GC-FID technique, ethylbenzene and o-p/xylene are quantified individually whereas they are
 141 detected together at the same mass (m/z of C₈-aromatics = 107 Da) by PTR-MS.

142 **2.2. Experimental protocol**

143 To determine the sorption coefficients, a blank experiment referred to in the following as “No
 144 sink” is first performed using an empty chamber to evaluate the sink effect on the chamber
 145 walls. For the adsorption phase, the chamber is supplied with humidified air containing the
 146 targeted VOCs using a gas cylinder connected to a dilution system (*Gas Calibration Units,*
 147 *Ionicon analytick*). This dilution unit allows controlling the total flow rate and the humidity of

148 the VOC mixture. When the monitored concentrations reach an equilibrium during the
 149 adsorption phase, the desorption phase is triggered by supplying the chamber with zero air at
 150 the same flow rate and humidity, using a zero air generator (*Claind*) and a water bubbler
 151 connected to mass flow controllers (MKS). Three experiments are performed with empty
 152 CLIMPAQ and two are performed using the test material, under the same experimental
 153 conditions. The conditions of CLIMPAQ experiments are compared to FLEC experiments in
 154 Error! Reference source not found..

155 **Table 1: Comparison of experimental conditions between the CLIMPAQ and FLEC experiments**

| Parameters | | CLIMPAQ | FLEC | | |
|--|--------------|-----------------------|---------------------|----------|----------|
| Temperature (°C) | | 23 ± 2 | 23 ± 2 | | |
| Relative humidity (%RH) | | 50 ± 5 | 50 ± 5 | | |
| Volume (V ; m ³) | | 50.9 10 ⁻³ | 35 10 ⁻⁶ | | |
| Air flow rate (F ; ml min ⁻¹) | | 200 | 300 | 400 | 500 |
| Air exchange rate (Q ; h ⁻¹) | | 0.3* (±5%) | 514 | 686 | 857 |
| Air velocity at test material surface (v ; m s ⁻¹) | | 0.16* | 0.0106** | 0.0142** | 0.0177** |
| Area of test piece (A ; m ²) | | 0.099 | 0.0177 | | |
| Loading factor (L ; m ² m ⁻³) | | 1.94 | 506 | | |
| Detection system (Time resolution) | | GC-FID (30 min) | PTR-MS (2-20s) | | |
| Concentrations (µg m ⁻³) | Benzene | 182 | 106 - 479 | | |
| | Toluene | 658 | 377 - 1131 | | |
| | Ethylbenzene | 128 | 289 - 868 | | |
| | P-xylene | 253 | | | |
| | O-xylene | 127 | | | |

*Experimental measurements ; ** [26]

156

157 3. Theory and data treatment

158 SigmaPlotTM and a Microsoft Excel Eulerian solver are used to extract sorption parameters
 159 from experimental data using three different models as detailed below.

160 The model used in the companion paper to extract sorption parameters is the Tichenor Model
 161 [10] referred in the following as “Tichenor Model - 1 surface” or TM-1S. This model, as
 162 described in the companion paper is suitable when only one sorption surface is considered
 163 (sorption effect on the chamber walls are negligible). However, as mentioned above, some
 164 studies have reported that for chambers of several liters, the chamber can itself act as a sink
 165 for some VOCs [16-18]. To account for this sink effect on the chamber walls and the tested
 166 material, the Tichenor Model is modified to be used when two sorption surfaces should be
 167 considered (Tichenor Model - 2 surfaces or TM-2S). Finally, as already mentioned in the
 168 introduction, the mass transport process taking place between the material surface and the
 169 bulk air should be taken into account to analyse chamber experiments. Thus, a model called
 170 “Tichenor Model with a boundary layer” or TM-BL combining the sorption and the mass
 171 transport processes is proposed.

172 3.1. Tichenor Model - 1 surface (TM-1S)

173 The Tichenor model, derived from the Langmuir theory [10] is based on the assumption that
 174 the concentration of a species in bulk air is homogeneous and proportional to the surface
 175 concentration. Therefore, the concentration variations (dC_g/dt and dC_s/dt) observed in a
 176 chamber depends on the adsorption (k_a) and desorption (k_d) coefficients, as shown in the
 177 following equations:

$$178 \quad \frac{dC_g}{dt} = -\frac{A}{V}(k_a C_g - k_d C_s) + \frac{Q}{V}(C_{in} - C_g) \quad (1)$$

$$179 \quad \frac{dC_s}{dt} = k_a C_g - k_d C_s \quad (2)$$

180 where, C_{in} is the VOC concentration at the chamber inlet performed ($\mu\text{g}\cdot\text{m}^{-3}$), C_g the gas-
 181 phase VOC concentration inside the chamber ($\mu\text{g}\cdot\text{m}^{-3}$), A is the material surface (m^2), V the
 182 volume of the chamber, Q the air flux in the chamber ($\text{m}^3\cdot\text{h}^{-1}$), C_s the surface concentration
 183 ($\mu\text{g}\cdot\text{m}^{-2}$), k_a the adsorption rate constant ($\text{m}\cdot\text{h}^{-1}$) and k_d the desorption rate constant (h^{-1}).

184 3.2. Tichenor Model - 2 surfaces (TM-2S)

185 The model TM-2S is used to extract VOC sorption parameters on the material surface taking
 186 into account the sorption effect on the chamber walls. Equations 1 and 2 represents the model
 187 TM-2S when the number of materials is $n=2$.

$$188 \quad \frac{dC_{g,i}}{dt} = -\sum_{j=1}^n \frac{A_j}{V} (k_{a,ij} C_{g,i} - k_{d,ij} C_{s,ij}) + \frac{Q}{V} (C_{in,i} - C_{g,i}) \quad \forall i \quad (3)$$

$$189 \quad \frac{dC_{s,ij}}{dt} = k_{a,ij} C_{g,i} - k_{d,ij} C_{s,ij} \quad \forall i, \forall j = 1, n \quad (4)$$

190 where, $C_{g,i}$ is the concentration of a pollutant i in the chamber gas phase ($\mu\text{g}\cdot\text{m}^{-3}$), A_j is the
 191 surface of a material j (m^2), V is the volume of the chamber (m^3), Q is the air flux in the
 192 chamber ($\text{m}^3\cdot\text{h}^{-1}$), $C_{in,i}$ is the concentration of a pollutant i at the inlet of the chamber ($\mu\text{g}\cdot\text{m}^{-3}$),
 193 $C_{s,ij}$ is the concentration of a pollutant i on the surface of a material j ($\mu\text{g}\cdot\text{m}^{-2}$), $k_{a,ij}$ is the
 194 adsorption coefficient of a pollutant i on the surface of a material j , and $k_{d,ij}$ is the desorption
 195 coefficient of a pollutant i on the surface of a material j .

196 3.3. Tichenor Model with a boundary layer (TM-BL)

197 This model TM-BL is applied to three compartments in the chamber: the material surface, the
 198 boundary layer (i.e. a thin layer of air exhibiting a concentration gradient above the material
 199 surface) and the bulk air above the material. The mathematical equations to model the
 200 concentrations in each compartment are detailed below.

$$201 \quad V \frac{dC_{g,i}}{dt} = \sum_{j=1}^n h_{m,ij} A_j (C_{ij}^* - C_{g,i}) + Q(C_{in,i} - C_{g,i}) \quad \forall i \quad (5)$$

$$202 \quad V_{ij}^* \frac{dC_{ij}^*}{dt} = h_{m,ij} A_j (C_{g,i} - C_{ij}^*) - A(k'_{a,ij} C_{ij}^* - k'_{d,ij} C_{s,ij}) \quad \forall i, \forall j = 1, n \quad (6)$$

$$203 \quad \frac{dC_{s,ij}}{dt} = k_{a,ij} C_{ij}^* - k_{d,ij} C_{s,ij} \quad (7)$$

204 where, C_{ij}^* is the concentration of a pollutant i in the elementary layer of air present above the
 205 surface of the material j and which defines the limit between the boundary layer and the
 206 surface of the material ($\mu\text{g m}^{-3}$), V_{ij}^* is the volume of the elementary layer of air present above
 207 the surface of the material j (m^3) and $h_{m,ij}$ is the average mass transfer coefficient of a
 208 pollutant i acting on the surface of a material j (m h^{-1}). The coefficient h_m can be calculated
 209 (See [32]) based on equations provided by [33]. The boundary layer conditions depend on air
 210 velocity at the surface j and on the targeted compound i .

211 Assuming that $V_{ij}^* \ll V$, equation 4 for each species and surface becomes:

$$212 \quad C_{ij}^* = \frac{h_{m,ij} C_{g,i} + k_{d,ij} C_{s,ij}}{h_{m,ij} + k_{a,ij}} \quad (8)$$

213 When equation (6) is introduced into equations (3) and (5), they become:

$$214 \quad V \frac{dC_{g,i}}{dt} = \sum_{j=1}^n A_j \left(-\frac{k_{a,ij} h_{m,ij}}{h_{m,ij} + k_{a,ij}} C_{g,i} + \frac{k_{d,ij} h_{m,ij}}{h_{m,ij} + k_{a,ij}} C_{s,ij} \right) + Q(C_{in,i} - C_{g,i}) \quad \forall i \quad (9)$$

$$215 \quad \frac{dC_{s,ij}}{dt} = \frac{k_{a,ij} h_{m,ij}}{h_{m,ij} + k_{a,ij}} C_{g,i} - \frac{k_{d,ij} h_{m,ij}}{h_{m,ij} + k_{a,ij}} C_{s,ij} \quad (10)$$

216 Using the new formalism:

$$217 \quad k'_{a,ij} = \frac{k_{a,ij} h_{m,ij}}{h_{m,ij} + k_{a,ij}} \quad (11) \quad \text{and} \quad k'_{d,ij} = \frac{k_{d,ij} h_{m,ij}}{h_{m,ij} + k_{a,ij}} \quad (12)$$

218 The equations (7) and (8) have the same appearance than equation (1) and (2), with $k'_{a,ij}$ and
 219 $k'_{d,ij}$ being apparent sorption coefficients measured in a test emission chamber or in a real
 220 room, considering the influence of the mass transfer coefficient $h_{m,ij}$ due to the boundary
 221 layer.

$$222 \quad V \frac{dC_{g,i}}{dt} = \sum_{j=1}^n A_j (-k'_{a,ij} C_{g,i} + k'_{d,ij} C_{s,ij}) + Q(C_{in,i} - C_{g,i}) \quad \forall i \quad (13)$$

$$223 \quad \frac{dC_{s,ij}}{dt} = k'_{a,ij} C_{g,i} - k'_{d,ij} C_{s,ij} \quad (14)$$

224 **4. Results**

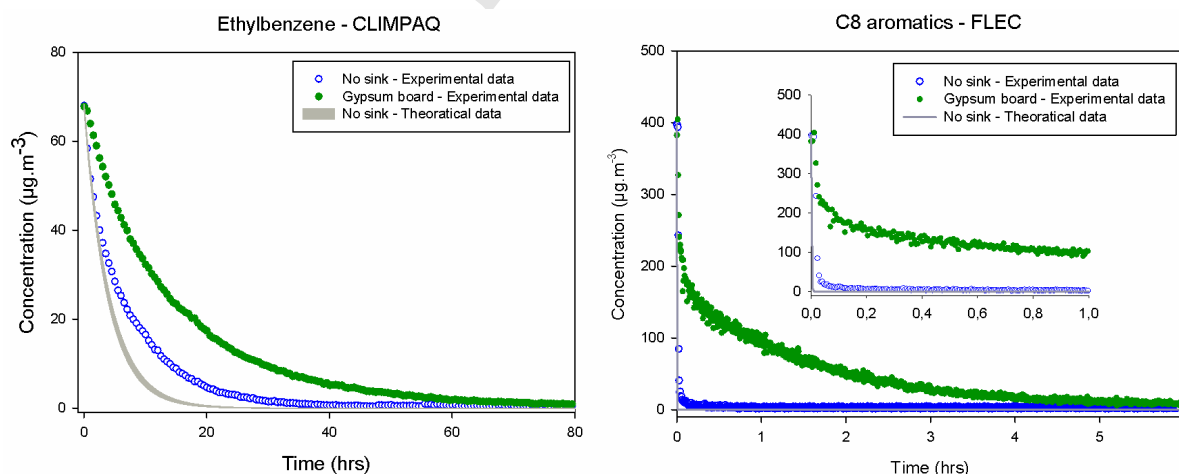
225 4.1. Sorption experiments in CLIMPAQ

226 The experimental concentration profiles obtained for the three experiments performed in the
 227 empty emission test chamber (No Sink) show relatively good overlay as well as the two
 228 experiments performed with the gypsum board (See Figure 1 in [32]). Therefore and for
 229 simplicity reason, only one profile for each type of experiment will be shown in the following.
 230 These profiles show that the duration of a sorption experiment in CLIMPAQ varies between
 231 20 and 40 hours for the No Sink and between 40 and 80 hours for the gypsum board,
 232 depending on the target VOC. However, the FLEC experiment is 6 to 13 times faster,
 233 showing the advantage that provides the FLEC-PTRMS method for field studies.

234 4.2. Investigation of VOCs sorption effect on chamber walls

235 The sink effect on the chamber walls is investigated by calculating the theoretical
 236 concentrations that should be obtained for a blank experiment (No sink) under the conditions
 237 of this study and assuming negligible walls effects. The equation used, accounts only for air
 238 exchange in the chamber and is $C(t) = C_e e^{-Nt}$ [10], with $C(t)$ the concentration versus time (μg
 239 m^{-3}), C_e the equilibrium concentration reached at the end of the adsorption phase (μg m^{-3}), and
 240 N the air exchange rate measured experimentally (h^{-1}).

241 Unlike the FLEC, the CLIMPAQ experiment shows a significant difference between the
 242 theoretical and experimental no sink profiles as shown in **Error! Reference source not found.**
 243 This difference is likely due to a sorption effect on the internal walls of the CLIMPAQ
 244 chamber and it is only observed for ethylbenzene and xylenes (See Figure 2 in [32]).
 245 Therefore the CLIMPAQ itself acts as a sink and the chamber surface should be considered in
 246 the analysis of the concentration profiles. VOCs sorption coefficients for ethylbenzene and
 247 xylenes on the chamber walls are assessed using the model TM-1S and obtained values for the
 248 three experiments performed in empty CLIMPAQ are presented in Table 2.



249
 250 **Figure 2. Comparison of concentration profiles obtained during the desorption phase for**
 251 **ethylbenzene using the CLIMPAQ method and C8 aromatics using the FLEC/PTRMS method.**

252 **Table 2. Obtained results for adsorption (k_a), desorption (k_d) and equilibrium (K_e) coefficients of**
 253 **ethylbenzene and xylenes on the CLIMPAQ walls using TM-1S. RSD is the random standard**

254 deviation corresponding to 1σ obtained for the three experiments referred as Exp.1, Exp. 2 and
 255 Exp.3.

| VOC | Sorption coefficient – Empty chamber | | | |
|--------------|--------------------------------------|--------------------------|-----------------------|------|
| | k_a (m.h ⁻¹) | k_d (h ⁻¹) | K_e (m) | |
| Ethylbenzene | Exp. 1 | $5,40 \times 10^{-3}$ | $2,16 \times 10^{-1}$ | 0,03 |
| | Exp. 2 | $6,84 \times 10^{-3}$ | $1,91 \times 10^{-1}$ | 0,04 |
| | Exp. 3 | $6,48 \times 10^{-3}$ | $1,87 \times 10^{-1}$ | 0,03 |
| | RSD (%) | 12 | 7,9 | 19 |
| O-Xylene | Exp. 1 | $5,04 \times 10^{-3}$ | $1,91 \times 10^{-1}$ | 0,03 |
| | Exp. 2 | $5,54 \times 10^{-3}$ | $1,62 \times 10^{-1}$ | 0,03 |
| | Exp. 3 | $5,40 \times 10^{-3}$ | $1,80 \times 10^{-1}$ | 0,03 |
| | RSD (%) | 4,9 | 8,2 | 13 |
| P-Xylene | Exp. 1 | $5,15 \times 10^{-3}$ | $1,66 \times 10^{-1}$ | 0,03 |
| | Exp. 2 | $7,20 \times 10^{-3}$ | $1,62 \times 10^{-1}$ | 0,04 |
| | Exp. 3 | $5,04 \times 10^{-3}$ | $1,37 \times 10^{-1}$ | 0,04 |
| | RSD (%) | 21 | 10 | 18 |

256 Obtained results show that the adsorption k_a and desorption coefficients k_d for ethylbenzene
 257 and o-p/xylene are in the same order of magnitude and presented consistent results between
 258 the three experiments with an RSD varying between 7.9 to 21%.

259 4.3. Determination of VOCs sorption parameters on test material

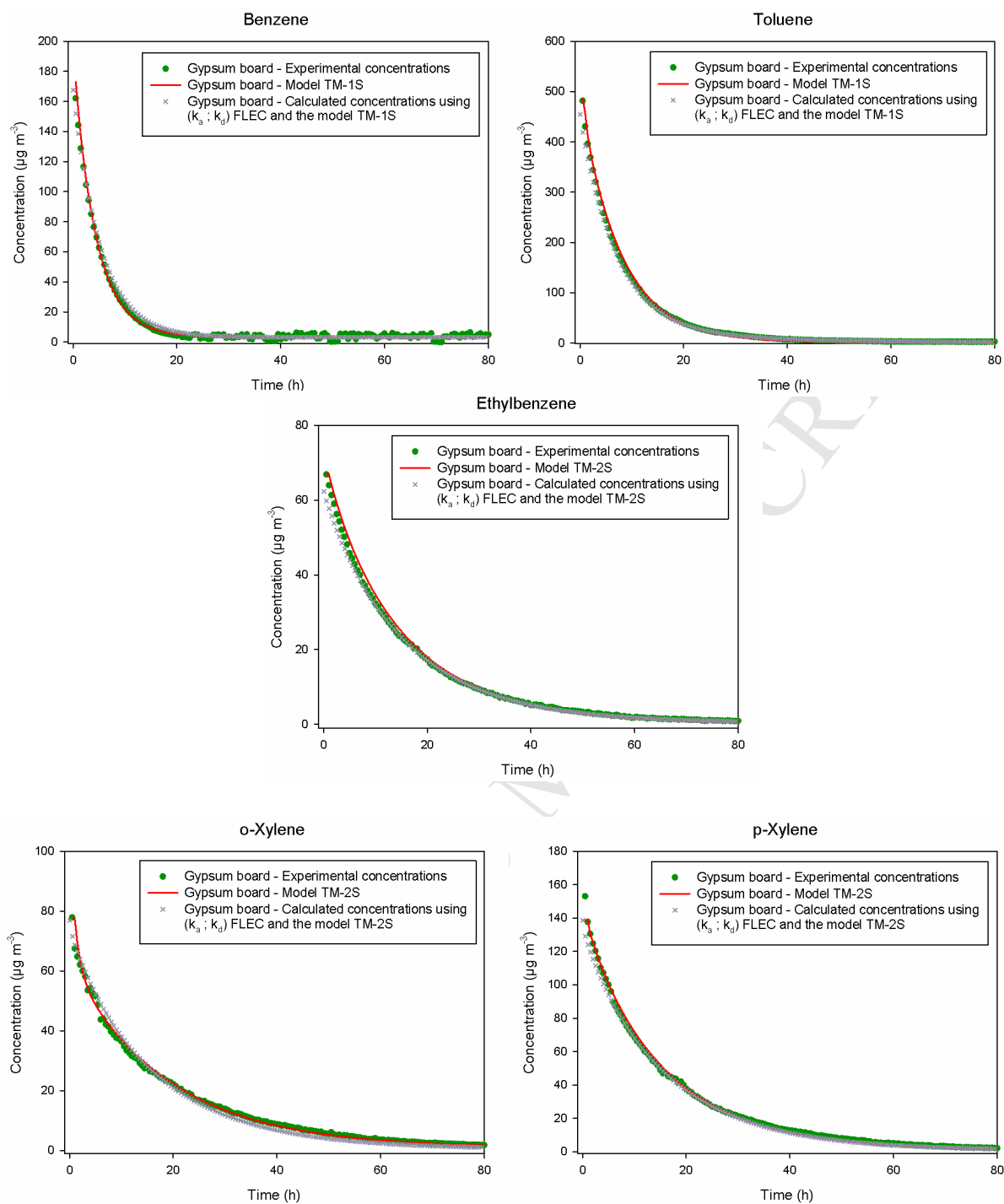
260 After the assessment of the sorption behaviour on the CLIMPAQ walls, sorption coefficients
 261 of the ethylbenzene and the o-p/xylene on the gypsum board are determined using the model
 262 TM-2S. However, the sorption coefficients of the benzene and toluene on the gypsum board
 263 are evaluated using the model TM-1S since any sorption effect on CLIMPAQ walls is noticed
 264 for these two compounds. Sorption coefficients of target VOCs on the Gypsum board are
 265 compared to those previously obtained with FLEC method (Part 1) in Table 3.

266 **Table 3. Comparison of BTEX sorption coefficients on gypsum board between FLEC and**
 267 **CLIMPAQ methods. n=5 correspond to five repeatability tests performed with the FLEC.**

| VOC | Model | Method | Sorption coefficients – Gypsum Board | | | |
|--------------|-------|------------|--------------------------------------|--------------------------|-----------------|-------|
| | | | k_a (m.h ⁻¹) | k_d (h ⁻¹) | K_e (m) | R^2 |
| Benzene | TM-1S | FLEC (n=5) | $1,5 \pm 0,13$ | $11 \pm 0,68$ | $0,13 \pm 0,01$ | 0,96 |
| | | CLIMPAQ | $3,25 \times 10^{-3}$ | 0,26 | 0,01 | 0,99 |
| Toluene | TM-1S | FLEC (n=5) | $1,5 \pm 0,10$ | $5,1 \pm 0,54$ | $0,30 \pm 0,04$ | 0,97 |
| | | CLIMPAQ | 0,33 | 1,02 | 0,32 | 0,99 |
| C8 aromatics | TM-1S | FLEC (n=5) | $1,3 \pm 0,18$ | $1,7 \pm 0,29$ | $0,76 \pm 0,12$ | 0,97 |
| Ethylbenzene | | | 14 | 18 | 0,80 | 0,99 |
| O-Xylene | TM-2S | CLIMPAQ | 0,16 | 0,20 | 0,79 | 0,99 |
| P-Xylène | | | 1,8 | 2,2 | 0,81 | 0,99 |

268 First, the coherence in the K_e coefficient results obtained with the CLIMPAQ chamber for
 269 ethylbenzene and o-p/xylene, proves the similar sorption behaviour for these 3 compounds.
 270 Thus, the comparison of their sorption behaviour can be done with the C8 aromatics (i.e. the
 271 sum of these 3 compounds when the FLEC-PTRMS method was used).

272 Additionally, the K_e ratios obtained for all tested compounds between both methods show a
273 good agreement except for benzene. Benzene shows a weak sorption effect on CLIMPAQ
274 walls, but also on the gypsum board and the determination of the sorption parameters is
275 consequently associated to a more important uncertainty than other compounds. However, the
276 adsorption and desorption parameters (k_a ; k_d) are significantly different between the
277 experiments conducted in the CLIMPAQ chamber and the FLEC. Given this high discrepancy
278 between k_a and k_d parameters determined with FLEC and CLIMPAQ methods, the FLEC
279 results obtained previously (k_a ; k_d) were used as inputs to simulate the concentration profile
280 that should be observed in the CLIMPAQ test chamber under the chamber operating
281 conditions and using the model TM-1S for the benzene and toluene and the model TM-2S for
282 the other compounds. Obtained profiles with FLEC data as shown on Figure 3, retrieves the
283 experimental profile as well as the model TM-1S used to retrieve sorption parameters for
284 benzene and toluene from the experimental profiles and the model TM-2S used for other
285 compounds. This results highlight the presence of several pairs of solutions (k_a ; k_d) that can
286 reproduce the same experimental profile obtained in CLIMPAQ.



287

288

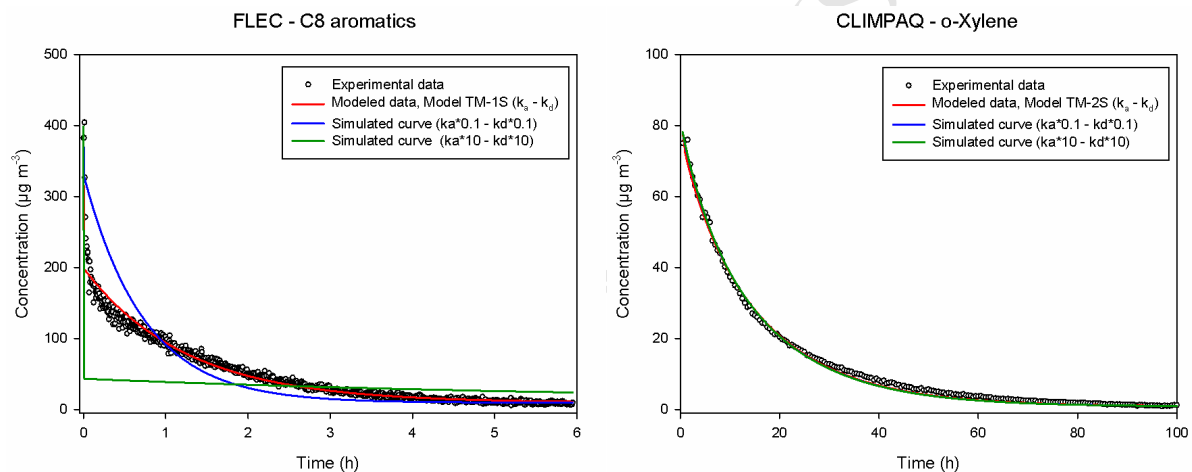
289

290 **Figure 3. Analysis of the experimental profile obtained for the gypsum board in the CLIMPAQ test**
 291 **chamber. The concentration profile obtained theoretically in CLIMPAQ using sorption parameters**
 292 **determined in FLEC is also presented.**

293

294 **5. Discussion**295 **5.1. Methods sensitivity**

296 Given the discrepancy between FLEC and CLIMPAQ methods in the determination of the
 297 adsorption and desorption parameters (k_a ; k_d), a further investigation is conducted to
 298 understand how many couples of (k_a ; k_d), could describe the same experimental concentration
 299 profile. A set of desorption curves are simulated for both the FLEC and CLIMPAQ apparatus,
 300 using different couples of (k_a ; k_d), but having the same K_e ratio. A factor α varying between
 301 0.01 and 100 is used to multiply both sorption parameters (αk_a ; αk_d). An example of
 302 simulated curves is displayed on Figure 4 for C8 aromatics and o-xylene for the FLEC and
 303 CLIMPAQ apparatus, respectively. Only 2 values of α (5 and 10) are shown in this figure.
 304 While a clear difference can be observed between the experimental profile and the simulated
 305 curves for the FLEC experiment, this difference is less significant for the CLIMPAQ
 306 chamber.



307

308 **Figure 4: Results obtained for simulated curves using a factor α for o-xylene using the CLIMPAQ**
 309 **chamber and for C8 aromatics using the FLEC.**

310 The difference between the simulated curve for each value of α and the experimental
 311 observations could be computed using a parameter Q defined below.

$$312 \quad Q_{\alpha} = \frac{\sum \Delta^2 (Exp - Mod_{\alpha})}{\sum \Delta^2 (Exp - Mod_{BF})} \quad (15)$$

313 $\sum \Delta^2 (Exp - Mod_{\alpha})$ is the sum of the square differences calculated between the experimental
 314 profile and the curve simulated using (αk_a ; αk_d) and $\sum \Delta^2 (Exp - Mod_{BF})$ is the sum of the square
 315 differences calculated between the experimental profile and the curve obtained from the best
 316 fit (BF) for (k_a ; k_d) when $\alpha=1$ (Modeled data on Figure 4).

317 Therefore, when the Q parameter presents a value close to 1, the difference between the curve
 318 simulated with (αk_a ; αk_d) and the modelled one (k_a ; k_d) is negligible. The curves presented on
 319 the Figure 4 show in the FLEC case a Q value of 5.6 and 16 for the curves simulated with ($5k_a$
 320 ; $5k_d$) and ($10k_a$; $10k_d$) respectively. However, in the CLIMPAQ case the Q parameter

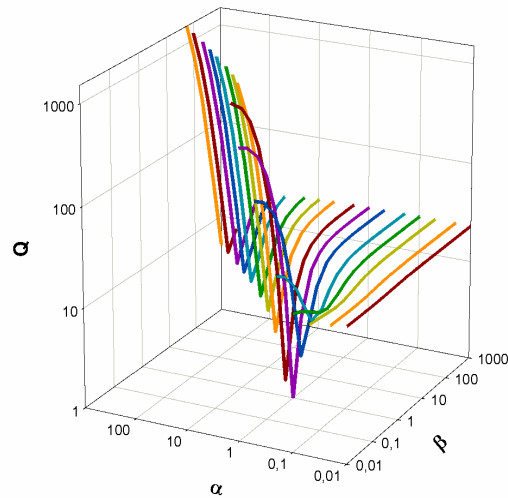
321 presents a value of 0.94 and 0.95 for the curves simulated with $(5k_a ; 5k_d)$ and $(10k_a ; 10k_d)$
322 respectively. Giving these results, the experimental concentration profile obtained using
323 CLIMPAQ can be retrieved using different couples of sorption parameters $(\alpha k_a ; \alpha k_d)$ having
324 the same K_e .

325 In order to look for all possible solutions, a large study is conducted using the same
326 methodology for different couples of $(\alpha k_a ; \beta k_d)$. It is worth noting that this time both k_a and
327 k_d are scaled by different factors α and β , respectively. This methodology is applied for all
328 sorption parameters determined for all compounds, using both CLIMPAQ and FLEC
329 methods, and for sorption on internal walls or on gypsum board.

330 First, the analysis result obtained for a “No sink” experiment performed in empty CLIMPAQ
331 is presented on Figure 5, for only the ethylbenzene and the o-p/xylenes since the toluene and
332 the benzene doesn't show any sorption properties on internal walls. This figure shows that a
333 single pair of solution (for $\alpha=\beta=1$) corresponds to $Q=1$. This result indicates that for no sink
334 experiments, the analysis of the experimental CLIMPAQ data using the model TM-1S, gives
335 only one value of $(k_a ; k_d)$.

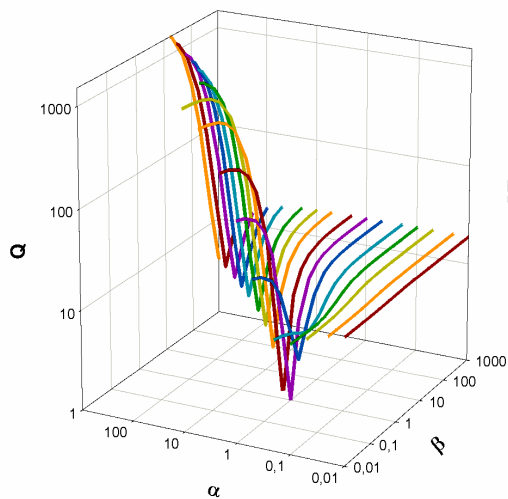
336

Ethylbenzene - CLIMPAQ - No Sink

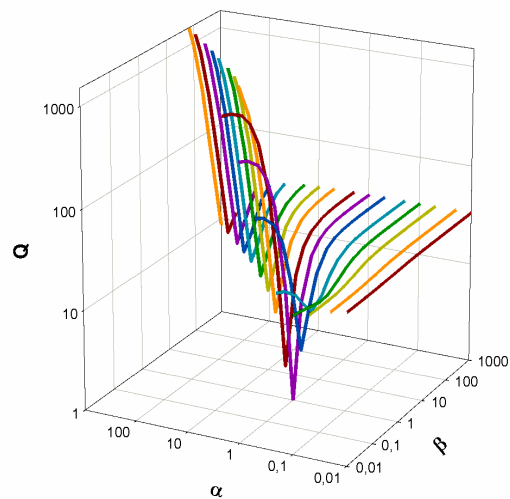


337

o-Xylene - CLIMPAQ - No Sink



p-Xylene - CLIMPAQ - No Sink

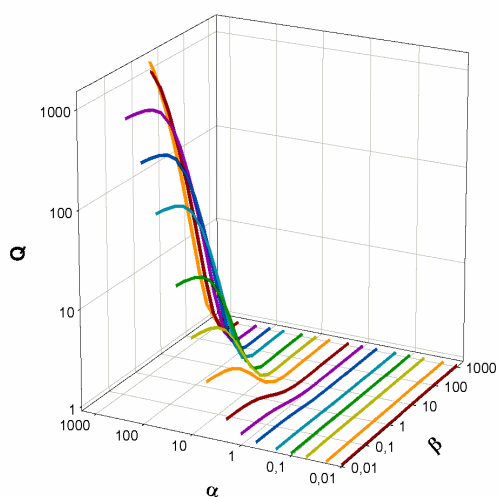


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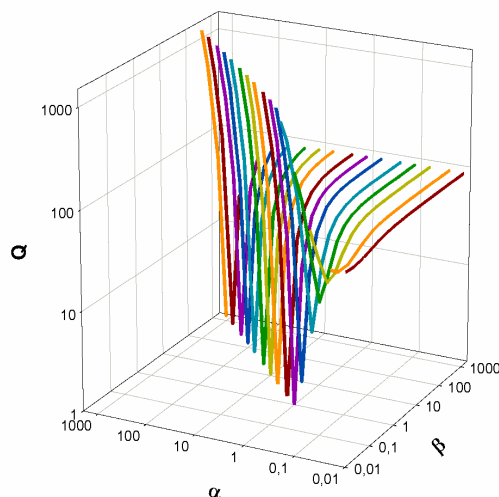
339 **Figure 5. Variation of the parameter Q as a function of the factors α and β used to multiply the**
 340 **sorption parameters k_a and k_d , in the case of empty CLIMPAQ chamber.**

341 Second, the same analysis performed for the sorption parameters determined for gypsum
 342 board using the CLIMPAQ method, is displayed on Figure 6. This figure shows that more
 343 than one couple of sorption parameters are characterized by a Q value close to 1 and
 344 corresponding to $\alpha=\beta$ (the model TM-1S is used for the benzene and toluene and the model
 345 TM-2S for the ethylbenzene and o-p/xylene). This result proves that different couples of
 346 solutions having the same K_e ratio lead to overlapped concentration profiles and
 347 consequently, the analyses of experimental data, may give several couples of solutions. It is
 348 important to note that the benzene presents a particular behaviour as shown on Figure 6 where
 349 many couples of sorption parameters (k_a ; k_d), not corresponding to the same K_e ratio, can
 350 retrieve the same concentration profile. This result can be due to the low sorption properties
 351 of benzene on gypsum board associated to an important uncertainty in the determination of its
 352 sorption parameters as already highlighted using the FLEC method.

Benzene - CLIMPAQ - Gypsum board

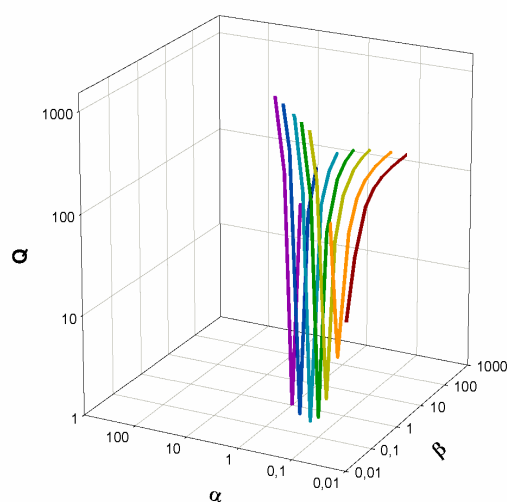


Toluene - CLIMPAQ - Gypsum board



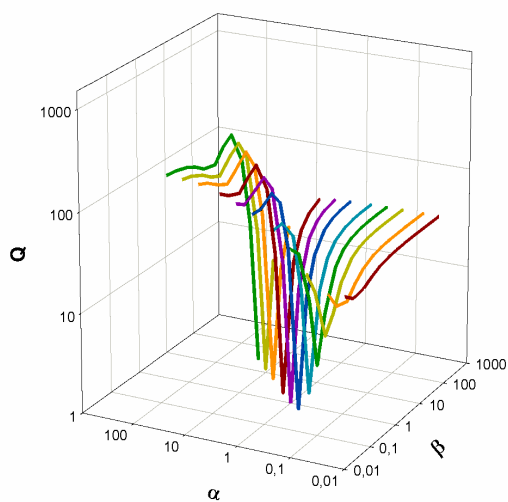
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Ethylbenzene - CLIMPAQ - Gypsum board

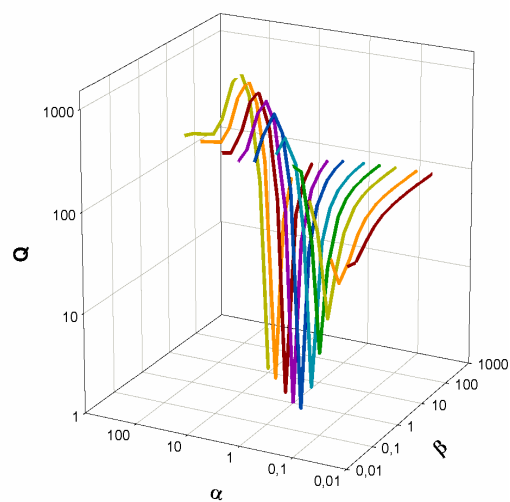


354

o-Xylene - CLIMPAQ - Gypsum board



p-Xylene - CLIMPAQ - Gypsum board



355

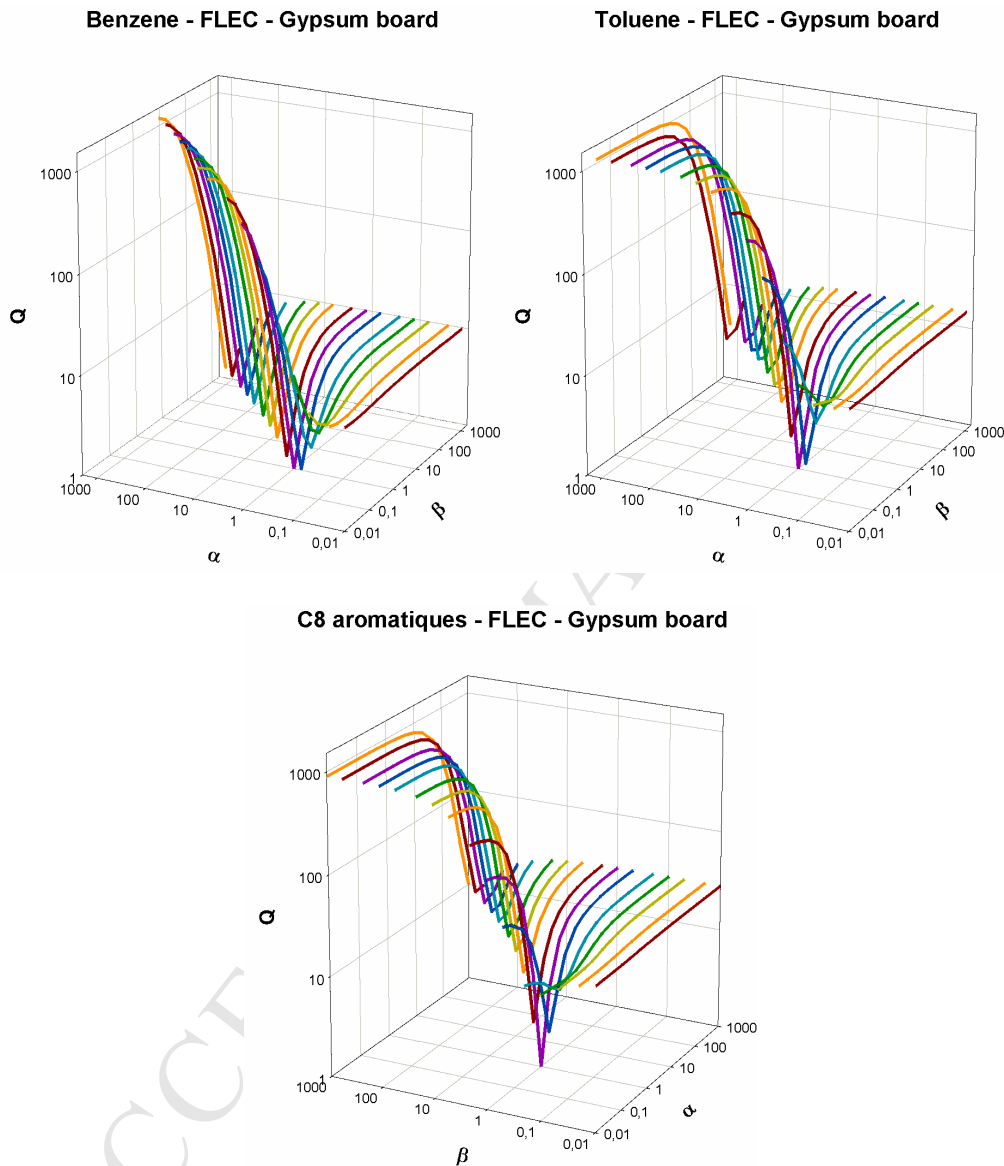
356

357

Figure 6. Variation of the parameter Q as a function of the factors α and β used to multiply the sorption parameters k_a and k_b in the case of gypsum board tested in a CLIMPAQ chamber.

358 Finally, in comparison to the FLEC method for a sorption experiment conducted on the
 359 gypsum board, Figure 7 shows that the Q value is found to be 1 for only one couple of
 360 sorption parameters, showing a unique solution $(k_a ; k_d)$ with $\alpha=\beta=1$. This result proves that
 361 the application of the model TM-1S on the experimental data, gives only one value of $(k_a ; k_d)$.

362



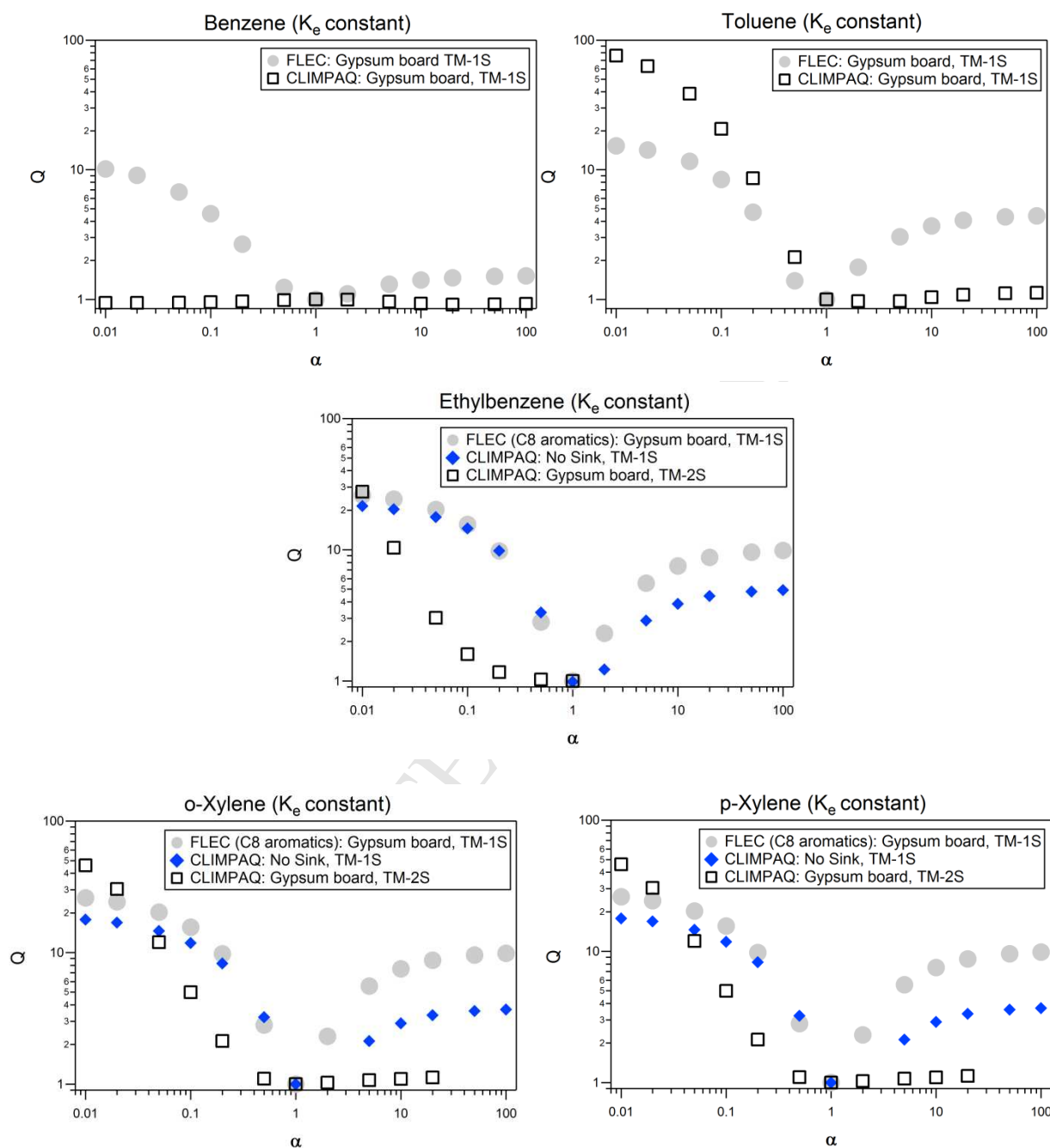
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364

365 **Figure 7. Variation of the parameter Q as a function of the factors α and β used to multiply the**
 366 **sorption parameters k_a and k_b in the case of gypsum board tested in a FLEC cell.**

367 In conclusion, Figure 8 presents the same analysis presented previously on Figure 5, 6 and 7,
 368 with a focus on the results obtained for a constant K_e ($\alpha k_a ; \alpha k_d$). In the FLEC case, Q is equal
 369 to 1 for only the couple of $(k_a ; k_d)$ determined by applying the model TM-1S to the
 370 experimental profile obtained for the gypsum board. Q increases significantly when different
 371 couple of $(k_a ; k_d)$ are used even if K_e is kept constant, which is consistent with the simulated
 372 curves being significantly different from experimental observations as shown in Figure 4.
 373 However, the parameter Q is close to 1 for the CLIMPAQ and remains at a constant value

374 when α varies between 1 and 100. Consequently, for the CLIMPAQ case, there is no
 375 significant difference between simulated curves using different (k_a ; k_d), if the ratio K_e is kept
 376 constant (Figure 4). It is interesting to note that a significant difference was observed only
 377 between both methods when the ratio α varies between 0.01 and 1.



381 **Figure 8 : Calculated ratio Q (Log scale) between experimental data and modeled curves obtained**
 382 **using different couples of sorption parameters (αk_a ; βk_d) for CLIMPAQ and FLEC.**

383 To conclude about the effect of the chamber size on the results observed above, some
 384 simulations are performed by changing the initial values of k_a and k_d with an factor α varying
 385 between 0.01 and 100, using three chambers having different volumes, 40 mL, 1 m³ and 30
 386 m³ (See Table 1 and Figure 3 in [32]). Only the micro chamber shows sensitive results for the

387 sorption parameters changed as already observed for the FLEC. However other chambers
 388 show similar results as the CLIMPAQ chambers. Therefore, small chambers, characterised by
 389 high loading factor and high air exchange rates like FLEC and the micro chamber are
 390 recommended to perform sorption experiments and to extract robust couples of solutions (k_a ;
 391 k_d) from an experimental profile while larger chambers seems only to provide robust K_e
 392 coefficients.

393 5.2. Influence of the mass transfer coefficient

394 The mass transfer coefficient h_m is calculated in the CLIMPAQ chamber under the conditions
 395 of this work from the correlations between dimensionless numbers that are the Sherwood
 396 number, Reynolds number and Schmidt number according to the equations presented in [32].
 397 According to Table 4, the flow of air is laminar in the CLIMPAQ chamber, since the
 398 boundary between the laminar flow and the transient state corresponds to a Reynolds number
 399 of 5×10^5 [34]. The mass transfer coefficient h_m varies between 3.1 and 3.4 $m\ h^{-1}$ depending
 400 on the type of VOCs. The thickness of the boundary layer developed on the surface of the
 401 material is estimated to be around 0.9 cm.

402 **Table 4. Parameters and dimensionless numbers used to calculate the mass transfer coefficient h_m**
 403 **in the CLIMPAQ test emission chamber.**

| | | Benzène | Toluène | Ethylbenzène | o-Xylène | p-Xylene |
|---|---|-----------------------|-----------------------|-----------------------|-----------------------|-----------------------|
| Molecular diffusivity of the binary VOC a in the air | $\alpha\text{-air}_D$ ($m^2.s^{-1}$) | 0,09 | 0,09 | 0,08 | 0,09 | 0,08 |
| Length of the surface in the direction of the air flow | L (m) | | | 0,60 | | |
| Kinematic viscosity of the air phase | ν ($m^2.s^{-1}$) | | | $1,57 \times 10^{-5}$ | | |
| Mean fluid velocity (parallel to the surface) outside of the boundary layer | U ($m.s^{-1}$) | | | 0,17 | | |
| Reynolds number | Re_L | | | $6,5 \times 10^3$ | | |
| Mass transfer coefficient | h_m ($m.s^{-1}$) | $9,52 \times 10^{-4}$ | $9,45 \times 10^{-4}$ | $8,56 \times 10^{-4}$ | $9,45 \times 10^{-4}$ | $8,70 \times 10^{-4}$ |
| | h_m ($m.h^{-1}$) | 3,4 | 3,4 | 3,1 | 3,4 | 3,1 |

404 According to the equations 9 and 10, the ratio K'_e could be represented by the equation (9):

$$405 \quad K'_e = \frac{k'_{a,ij}}{k'_{d,ij}} = \frac{k_{a,ij} h_{m,ij}}{k_{d,ij} h_{m,ij}} \quad (16)$$

406 where h_m is equivalent to the factor α used before to carry out the study of the methods
 407 sensitivity in the determination of sorption parameters. Nevertheless, the h_m value determined
 408 for the CLIMPAQ is in the range of variation of the factor α , corresponding to a couple of
 409 parameters (k'_a ; k'_d) that can reproduces the experimental trace as well as the couple ($k_{a,ij}$;
 410 $k_{d,ij}$). Therefore, the boundary layer effect cannot be investigated in the CLIMPAQ chamber
 411 under the conditions of this study, due to the presence of several possible solutions for the
 412 same experimental data.

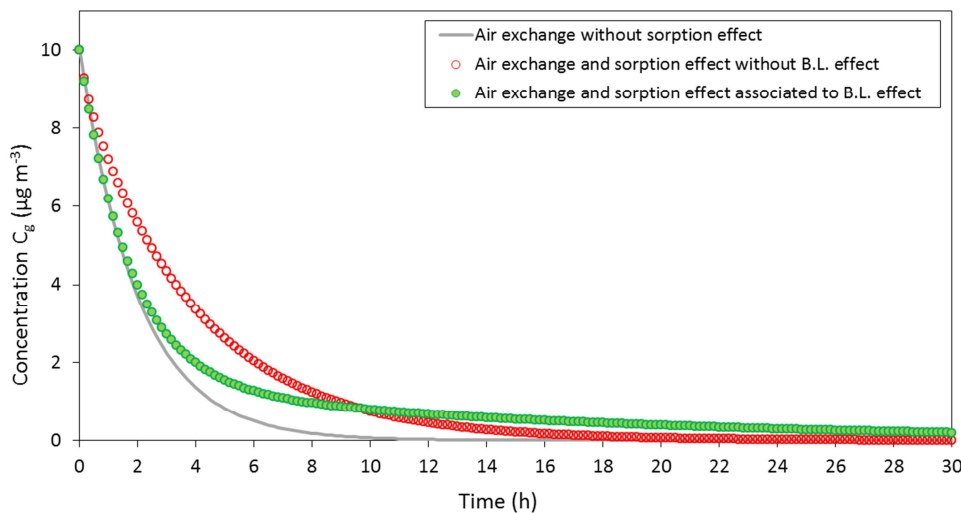
413 **5.3. Influence of the mass transfer coefficient in a real room**

414 The influence of the mass transport within the boundary layer is also investigated in a real
 415 indoor environment. For this, a real room of $7.2 \times 6.9 \times 2.7 \text{ m}$ ($V=134 \text{ m}^3$) is considered for the
 416 simulations. Ethylbenzene and a gypsum board material are selected as an example to model
 417 the concentration decay using 2 couples of sorption parameters. The first couple is the FLEC
 418 derived parameters ($k_{a \text{ FLEC}}$; $k_{d \text{ FLEC}}$) determined independently on the mass transfer
 419 coefficient h_m . The second couple used is (k'_a ; k'_d) calculated according to equations 11 and
 420 12, using FLEC results and the mass transport coefficient h_m calculated for the real room. All
 421 parameters used are shown in Table 5, where one air exchange rate (ACH) was chosen to
 422 mimic conditions when the ventilation is turned off. The details of all calculations are given in
 423 [32].

424 **Table 5: Main parameters used to model ethylbenzene concentration decays in a real room**
 425 **configuration.**

| Parameters | Real room parameters |
|---|----------------------|
| $C_g(0)$ ($\mu\text{g m}^{-3}$) | 10 |
| Volume, V (m^3) | 135 |
| Air exchange rate ACH, N (h^{-1}) | 0.5 |
| Surface of the material, S (m^2) | 150 |
| Loading factor, L ($\text{m}^2 \text{ m}^3$) | 1.11 |
| Length of the surface in the direction of the flow, l (m) | 7.6 |
| Air flow velocity, v (m.s^{-1}) | 9×10^{-4} |
| Mass transfer coefficient, h_m (m h^{-1}) | 0.07 |
| Thickness of the boundary layer, T_{BL} (m) | 4.1×10^{-1} |
| $k_{a \text{ FLEC}}$ (m h^{-1}) | 1.3 |
| $k_{d \text{ FLEC}}$ (h^{-1}) | 1.7 |
| k'_a (m h^{-1}) | 0.06 |
| k'_d (h^{-1}) | 0.08 |

426 ^a Ventilation off; ^b Ventilation on



427

428 **Figure 9: Decays of ethylbenzene concentration modeled in a real room taking into account air**
 429 **exchange and neglecting sorption effects (gray plot), taking into account sorption effects but**
 430 **neglecting the boundary layer effect ($k_a; k_d$) (red plot), and taking all processes into account**
 431 **($k'_a; k'_d$) (green plot).**

432 As expected, Figure 9 shows that the sorption effect leads to a slower decay of concentration
433 in a real room compared to the ventilation process. It is also interesting to note that the
434 concentration decay is significantly slower when the mass transport of VOC in the bulk air is
435 taken into account, which indicates that sorption processes on surfaces are significantly
436 influenced by the presence of the boundary layer on the scale of a real room. This result
437 highlights that sorption parameters should be used as data inputs to indoor air quality IAQ
438 models to predict concentrations in combination with a mass transport model. In the literature,
439 IAQ models usually considered indoor processes like emission, ventilation and chemical
440 reactions related to reactivity phenomena [35]. However, the interactions of pollutants with
441 surfaces through sorption processes were never before considered in IAQ models except the
442 newly developed INCA-indoor model [36]. Using the FLEC-PTRMS device, it is become
443 possible to measure VOC sorption parameters on indoor surfaces to validate this model and
444 predict better indoor air concentrations.

445 **6. Conclusion**

446 Comparing the equilibrium coefficient K_e , it was proved that the sorption properties assessed
447 are consistent for all VOCs using the FLEC and the CLIMPAQ methods. However, using the
448 CLIMPAQ method, several limitations can introduce biases in the determination of
449 adsorption and desorption parameters, k_a and k_d , respectively. The sink effect of the chamber
450 walls can introduce a bias on derived parameters if it is not taken into account in the
451 mathematical model used to analyze the concentration profiles. Giving the experimental error
452 of 15% observed on measured concentrations [31], several couples of (k_a , k_d) values can be
453 determined from the same set of experimental data with the CLIMPAQ chamber. In contrast,
454 it is found that the analysis of FLEC data is more sensitive to the variation of sorption
455 parameters, and as a consequence, the method seems more robust for measuring sorption
456 parameters. The robustness of the fit procedure was never assessed before in literature and the
457 discrepancy found can be behind the difference observed with indoor air quality models using
458 chamber derived parameters.

459 In actual applications conducted in our group, the FLEC method proved to be promising, very
460 useful for both field and laboratory experiments even if it requires a fast analytical tool such
461 as the PTR-MS. The measurement of sorption parameters can be performed within a few
462 hours, and a simple model can be used to accurately derive the elementary sorption
463 coefficients of several VOCs on the surface of homogenous materials. In addition, the TM-BL
464 model used in this study is well adapted to use these data in combination with the VOC mass
465 transfer properties in the gas phase, to describe the influence of adsorption and desorption
466 processes on ambient concentrations. However, the influence of the diffusion in the inner of
467 the material is not taken into account since it is considered to be slower than the instantaneous
468 sorption phenomena on the surface of the tested material. In real case, it is interesting to
469 predict indoor air concentrations using a model that takes into account the diffusion inside
470 porous materials and the available quantity of VOCs inside a material present on the field.
471 Unfortunately, it is a real challenge to measure on the field such parameters [36] and further
472 works are needed to develop rapid and accurate method to determine the diffusion
473 characteristics in porous material on the field [19].

474 **Acknowledgments**

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 477 of Malak Rizk and MERMAID project under PRIMEQUAL program.

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Highlights

- First comparison of two methodologies used to measure VOC sorption parameters on indoor surfaces
- Highlighting on the robustness and the advantages of the FLEC-PTRMS method for field measurements
- Usefulness of FLEC derived parameters as data inputs for indoor air quality models