



# Efficiency and harmfulness of air-purifying photocatalytic commercial devices: From standardized chamber tests to nanoparticles release



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## ABSTRACT

The aim of this study was the comparison of the efficiency of several commercial photocatalytic air-purifiers according to the French XP B44-013 AFNOR standard test (soon European) in a large closed chamber at the ppbV level under controlled conditions representative of indoor air. After inter-lab comparisons validating the method and analytical procedures, the commercial devices were evaluated for the mineralization of a mixture of four representative VOCs (acetaldehyde, acetone, *n*-heptane and toluene). Comparison of the degradation rate allowed the determination of the clean air delivery rate (CADR), while the mineralization efficiency was determined from CO<sub>2</sub> analysis. The devices could be ranked in two classes: class 1 for efficient devices (high CADR, extended mineralization, no by-product) and class 2 for inefficient and unsafe ones (by-product release even in the absence of VOCs, low CADR and mineralization). Electrical low pressure impactor (ELPI) could not evidence any nanoparticles release with any of the studied devices. Formaldehyde was the main by-product detected in all cases, but the concentration remained low and decreased with time due to its total mineralization with class 1 devices. For class 2 devices, formaldehyde concentration was higher and went on increasing with time.

These experimental data point out the urgent need of careful evaluation and certification of commercial photocatalytic air-purifiers for consumer's safety and secure development of the technology.

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## 1. Introduction

Current concern for indoor air quality (IAQ) [1] provides opportunities for the development of photocatalytic oxidation (PCO) since it is a promising technology for the removal of volatile organic compounds (VOCs) at low ppbV concentration and low flow rate in indoor air at room temperature and atmospheric pressure [2–4]. However, most of the studies devoted to indoor pollution dealt with high pollutants concentrations (10–100 ppmV or part per million in volume) with single or binary mixture of model molecules, out of the range usually encountered for indoor air (10–1000 ppbV or part per billion in volume). Only few studies were carried out in the ppbV range, sometimes coupled with odor characterization [5–8]. Moreover, the comparison of the results obtained in different laboratories is often unreliable, since a lot of operating conditions (design of the reactor, irradiation source and light intensity, flow rate, pollutant nature and concentration, pure pollutant or mixture of them, relative humidity, etc.) influenced the fate

of the photocatalytic reactions. The analysis and determination of the intermediate products is not always complete, and one of the main indicators, i.e. the extent of mineralization, issued from the determination of CO<sub>2</sub> evolution during the reaction, is not always available.

It thus appears very important for the consumer's safety and for the secure development of the market to design reliable standards to assess the performances of commercial photocatalytic products and especially of air purifiers. Besides the already published ISO standards [9], the CEN TC 386 "photocatalysis" has been working at the European level since November 2008. Among the eight working groups (WGs), WG2 is now improving the French AFNOR XP B44-013 standard published in December 2009 for the evaluation in a closed-chamber test of the photocatalytic function of indoor air purifiers (Table S1). Several laboratories were involved in the development and assessment of this new standard (NORMACAT project), which led to consistent inter-laboratories comparison of a given device. The analysis of the photocatalytic reaction rate and products, including CO<sub>2</sub> to determine the mineralization yield, on a well-defined mixture of four VOCs (toluene, heptane, acetaldehyde, acetone) was followed with suitable but different analytical devices [10].

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The aim of the present study is the comparison of the performances of several commercial photocatalytic devices according to this closed-chamber test. Since the objective of the project is the appraisal of the efficiency and safety of these commercial devices, preliminary results on their aging as well as on the possible nanoparticles emission will be presented.

## 2. Materials and methods

The protocol used for photocatalytic closed-chamber test followed the AFNOR XP B44-13 standard. Comparison of the airtight chambers from CERTECH and IPREM with additional details were reported previously [10]. The validation of the 1.2 m<sup>3</sup> closed-chamber test (Fig. S1) was made before the photocatalytic experiments in order to control the leaks, the possible release of VOCs from the chamber materials and the stability of VOC concentration without photocatalytic device for 24 h (less than 10% variation). The chamber was equipped with an external fan (minimum flow 120 m<sup>3</sup> h<sup>-1</sup>), and a relative humidity and temperature probe. Online connection of the analytical systems (GC-FID, PID and FID-methanizer from AIRMOTEC for the determination of VOCs and CO/CO<sub>2</sub> respectively, Table S2) to the chamber allowed automatic sampling. For aldehydes analysis, the LpDNPH S10 Cartridges (Supelco) containing high-purity silica coated with 2,4-dinitrophenylhydrazine (DNPH) were used for sampling air. A pump sampled air from the device through the cartridge at 1 L min<sup>-1</sup> for 15–20 min. The cartridges were then desorbed in reverse direction to the airflow with 5 mL acetonitrile. Twenty microliters of solvent were injected into the HPLC system (Knauer). A formaldehyde curve of formaldehyde-DNPH was used (from commercial standard Sigma Aldrich) for the quantification of the content of formaldehyde in the air samples. For other secondary VOCs, possibly formed as intermediate products, Air Toxics or Tenax GR stainless steel tubes (Supelco) were used for sampling air at a flow of 0.1 L min<sup>-1</sup> for 10–15 minutes. The tubes were then desorbed in the reverse direction of the airflow using ATD coupled with GC-MS (Turbomatrix ATD650-GC-MS Clarus 680 PerkinElmer). During the photocatalytic test, the sampled air volume should always be kept under 5% of the chamber volume, i.e. 60 L maximum of sampled air. In all the cases, the ratio of the volume of the device to that of the chamber was less than 6% (and should be <25% according to the standard).

Before the test, the chamber was first flushed with clean dry air and the photocatalytic device switched on during 12 h to clean the photocatalytic media. In the second step, the chamber was flushed with clean moist air until the defined relative humidity (50 ± 5%) was obtained. A control (temperature, relative humidity, VOC and CO<sub>2</sub> data) with device on warranted the quality of the chamber/device, with no unexpected pollutant emission in the baseline. The photocatalytic device was then switched off. For the efficiency experiment, the standard mixture was added from pure liquid commercial solution (Sigma Aldrich or Acros) with a microsyringe through a septum into the chamber at two concentrations (250 and 1000 ppbv each compound). The external fan was switched on only during pollutants homogenization and switched off after homogenization, when the photocatalytic device started. The experiment was carried out until more than 90% VOC disappeared or during 24 h. Continuous VOC, CO<sub>2</sub> analysis, temperature and relative humidity evolution were followed before pollutants addition and during the complete photocatalytic test.

For micro- and nanoparticle release, a low-pressure cascade impactor (ELPI, DEKATI) was directly connected to the chamber and the air was sampled at a flow rate of 10 L min<sup>-1</sup>, with a corresponding inlet of fresh filtered air (Ultra-Filter Donaldson DF-P/S 0035 and filter RoHS AFD20-F02C) on another port of the chamber.

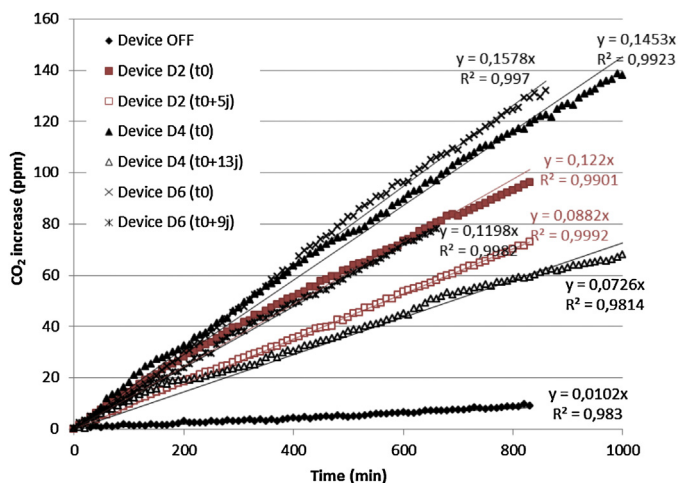


Fig. 1. CO<sub>2</sub> increase during two successive control tests without added VOC for D2, D4 and D6 within few days interval ( $T_0$  = first control test,  $T_{0+xj}$  = second control test after  $x$  days).

This device was used to measure on-line the particle size distribution from 10  $\mu$ m to 7 nm distributed over 13 stages with a time resolution of 10 s.

Eight commercial photocatalytic devices (D1–D8) were compared in the closed chamber with a CERTECH custom-made photocatalytic device (D0) previously used for inter-laboratories analyses. Some features of the commercial devices (from the providers) are reported in Tables 1 and 2: some of them (D1–D4) only had a photocatalytic function, while the others (D5–D8) also had an additional filter or function.

## 3. Results and discussion

### 3.1. Comparison of the efficiency of various photocatalytic devices: CADR calculation

In order to compare various photocatalytic devices with possible additional functions (ionization, filtration), two sets of data were analyzed: the decrease in VOCs concentration with time and the increase in CO<sub>2</sub> concentration with time indicative of the photocatalytic pollutants mineralization. For all the studied systems, the experiments were carried out successively without (control test to detect any VOC or CO<sub>2</sub> production by the device itself) and with VOC (photocatalytic test) in the closed chamber (Table 1).

For the control tests, VOC production was detected for three among the nine studied devices, namely D5, D7 and D8. The main VOCs released after 24 h in the closed chamber were acetaldehyde (<260 ppbv), acetone (<160 ppbv) and formaldehyde (<42 ppbv). Other released compounds from the device were also identified in lesser amounts by ATD-GC-MS of sampling cartridges (Table 1). This VOC release by these three devices implies that some pieces are not stable when switched on: either the photocatalytic material or some constituting components submitted to UVC (for D5 and possibly D8) or UVA (for D7) are emitting VOCs.

During these control tests without added VOC, CO<sub>2</sub> production was observed with five devices among the nine tested ones (Fig. 1). It should be noted that this CO<sub>2</sub> production decreased between the first and second runs carried out within a few days under the same experimental conditions (after flushing the chamber with clean moist air until the defined relative humidity (50 ± 5%) was obtained). This CO<sub>2</sub> production by the device itself may be accounted for by the mineralization of some organic volatile components emitted by the device. Because of the decrease of this production between the first and second runs, it may be assumed

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