### High concentration propane depletion with photocatalysis

C. Piferi<sup>1</sup> and C. Riccardi<sup>1, a)</sup>

Department of Physics, University of Milano-Bicocca, Piazza della Scienza 3, 20126, Milano, Italy

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Our work is aimed to study the volatile organic compounds (VOCs) abatement produced by photocatalysts working in different light spectrum, investigating the best catalyst able to treat propane in air mixtures at high concentrations of the order of thousands ppm. The experimental data were analyzed in order to extract the relevant parameters and to compare the catalytic activities of three different photocatalysts, TiO<sub>2</sub>, WO<sub>3</sub> and mixtures of them. In a reactor box of 1.5 L, a photocatalysis processing with TiO<sub>2</sub> catalyst gave the best propane depletion of the order of 10% for initial propane concentrations up to 5000 ppm after 22 minutes and UV-A lamps with an intensity of 0.4 mW/cm<sup>2</sup>. While the TiO<sub>2</sub> and WO<sub>3</sub> catalysts produced an abatement of about 5% after 22 minutes, using UV-C light at the intensity of 5  $10^2$  mW/cm<sup>2</sup>.

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<sup>&</sup>lt;sup>a)</sup>Electronic mail: claudia.riccardi@unimib.it.

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## I. INTRODUCTION Several methods have been proposed for VOCs depletion, including adsorption<sup>1</sup>, absorption<sup>2</sup>, thermal decomposition<sup>3</sup>, catalytic oxidation<sup>4</sup>, bio-filtration<sup>5</sup>, membrane separation<sup>6</sup>.

Photocatalytic oxidation (PCO) of VOCs is a very attractive and promising alternative technology for air purification<sup>7</sup>.

It has been demonstrated that organics can be oxidized to carbon dioxide, water and simple mineral acids at low temperatures on metal oxides catalysts using, for example, TiO2 catalyst. Several parameters affect the VOC conversion processes: the specific surface area of catalyst and the VOC concentrations. However the time conversion takes hours and is effective for low VOC concentrations of the order of tens of ppm. In addition is critical also the choice of the substrate to be degraded, its concentration and its significance in actual use, and the type of irradiation source and its irradiance at the sample surface.

For VOC degradation at relatively higher concentrations, of the order of hundreds and thousands ppm new studies have oriented to thermal plasmas like spark discharges<sup>8–10</sup> and Non-Thermal Plasmas NTP<sup>11,12</sup>. While thermal plasmas are too expensive, non-thermal plasma (NTP) has recently attracted more attention as a convenient and clean alternative.

In the non-thermal plasma chemical reactions charged species and radicals are produced in steady-state conditions. The primary electrons collide with background molecules producing secondary electrons, photons, ions and radicals<sup>13</sup>. This process is highly non-selective and creates a chemical reactive environment able to treat materials as well as gases at room temperature<sup>14–16</sup>. Previous studies on NTP demonstrated they are very effective in material applications by employing different gas precursors, because of its ability to highly dissociate molecules in gas phase, producing very reactive chemical groups at room temperature: materials are not damaged and can be easily functionalized<sup>17–20</sup>. When generated at atmospheric pressure plasma produces chemical species able to also dissociate VOC molecules at higher concentrations up to thousand ppm<sup>21–24</sup> but the processes are non selective.

Despite the success of NTP in VOC dissociation, the production of by-products

such as ozone, aerosol particle and incompletely oxidized compounds requires further research including chemical simulations in order to understand the process phenomenology<sup>25–27</sup>. A combination of NTP and catalysis were attempted in order to increase the depletion efficiency. Addition of catalyst bed near the plasma zone seems to enhance the decomposition rate of pollutants, reducing the undesirable by-products, due to the increase of retention time<sup>28</sup>.

In fact NTPs can contain a diverse mix of highly reactive species, they are difficult to operate in such a way as to produce single products in high yield and at high selectivity<sup>29,30</sup>. Integration of plasma and catalysts together promises to combine the advantages of the two, to effect transformations that are currently difficult or impossible to achieve<sup>31,32</sup>.

In our recent studies we were employing NTP surface discharges in which discharge is performed on the surface of the dielectric layer where also an electrode is located<sup>33–35</sup> In this plasma configuration, the catalysis can be integrated near the plasma source, directly near the electrodes.

In order to increase the abatement efficiency and to understand which mechanisms underlying the depletion processes some more research would be focused on the kinetics of the reaction products from an experimental point of view.

A better insight into the underlying physical and chemical processes is crucial and can be obtained by experiments applying diagnostics, studying both the chemical processes occurring at the catalyst surface and the dependence of the catalysis processes on the VOC concentration<sup>36,37</sup>.

Our work is aimed to the study of the abatement produced by photocatalysts working in different light spectrum, investigating the best catalyst able to treat propane in air mixtures at high concentrations. These studies represent the starting point for the next research in which catalyst and NTP will be combined in an hybrid reactor system<sup>38</sup>.

We performed an experimental reactor in order to study the VOC abatement by catalytic processes using  $TiO_2$ ,  $WO_3$  and their mixtures and UV-A, UV-C and visible light. We study the reaction kinetics in different propane gas air mixtures, evaluating the propane abatement as a function of time. The experimental data were analyzed in order to extract the relevant parameters and to compare the three

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TABLE I: Intensity emission of our lamps at different distances<sup>39</sup>.

	$0.5~\mathrm{cm}$	11 cm
Distance	(lamp inside	(lamp outside
	the box)	the box)
UV-A	$30 \ { m mW/cm^2}$	$0.4 \text{ mW/cm}^2$
UV-C	$4 \mathrm{W/cm^2}$	$35 \ \mathrm{mW}/\mathrm{cm}^2$
Visible	$0.5 \ { m W/cm^2}$	$0.9~{\rm mW/cm^2}$

photocatalytic activities.

We worked at high propane concentrations between 1200 and 5000 ppm, in view of further applications in which photocatalysis will be combine with plasma processing. In literature there are few experiments carried out by photocatalysis at these high concentrations. This work therefore intends to explore these conditions in order to broaden the effects of photocatalysis in this new application field.

### EXPERIMENTAL SETUP II.

In our experiment we used a vacuum cross chamber (20 cm length and 10 cm diameter) with a volume of about 1.5 l. The catalytic support are placed in a chamber and the lamps are placed faced to the catalytic support inside the chamber or outside the chamber in front of a quartz window. Three of the four opening are closed by vacuum gauges, the last one is closed by a quartz window to let the emission light from the lamp activate the catalyst (Figure 1).

The lamp characterization has been previously explained in our article<sup>39</sup>. The intensity emission of our lamps are reported in Table I.

We employed two different catalysts  $TiO_2$  and  $WO_3$ , in different mixtures deposited on plane rectangular tile surfaces. In this paper we refer to the following three depositions:

- A: 34.5 mg/cm<sup>2</sup> of TiO<sub>2</sub> deposited over 7.6x7.3 cm<sup>2</sup> tile;
- B: 40.9 mg/cm<sup>2</sup> of 50% TiO<sub>2</sub> + 50% WO<sub>3</sub> deposited over 7.7x7.5 cm<sup>2</sup> tile;

• C:  $38.2 \text{ mg/cm}^2$  of WO<sub>3</sub> deposited over  $7.4 \text{x} 7.3 \text{ cm}^2$  tile.

The setup is shown in Figure 2. The black bold lines represent the tube connections through the whole system. In the diagram, pure air represents a gas cylinder containing approximately 80% of N<sub>2</sub> and 20% of O<sub>2</sub>, meaning that the concentration of CO<sub>2</sub>, CO, hydrocarbons and other pollutants is negligible. The red crosses are the symbol of open-close valves. The VOC gas (propane) is placed in a cylinder and its flow is regulated by the flow meter 1, while the pure air cylinder flow is regulated by the flow meter 2. We used two gas flow meters by Bronkhorst factory.

For the gas chromatographic analysis we used a Micro GC Agilent 3000 instrument.

### III. MEASUREMENT RESULTS

### A. Gas chromatograph calibration

The gas chromatograph (GC) output reports a signal composed of peaks of intensity proportional to the concentration of specific species. The area of each peak is strongly dependent on the setting parameters during the sampling conditions. For a precise evaluation of concentration, it is necessary to perform first a specific calibration. Usually, the calibration is carried out using a cylinder containing a known concentration of the desired gas (one point calibration). However, we decided for a multipoint calibration.

As discussed in Sec. II, we deliver propane by means of a flow meter system (Figure 2). The propane flow relative to the total (air-pentane) input flow, denoted as c, typically expressed in ppm, is given by

$$c = \frac{\Phi_{C_3H_8}}{\Phi_{\rm air} + \Phi_{C_3H_8}},\tag{1}$$

where  $\Phi_{air}$  is the pure air flow and  $\Phi_{C_3H_8}$  is the propane flow, given in liters/sec. The values of air and propane flows are measured by the gas flow meters (Figure 2). These two values determine the initial conditions of the experiment before the treatment. The relative propane flow, Eq. (1), can be used to determine the actual inis is the author's peer reviewed, accepted manuscript. However, the online version of record will be different from this version once it has been copyedited and typeset PLEASE CITE THIS ARTICLE AS DOI: 10.1063/5.0073924

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propane concentration (in ppm), present in the air-propane mix system, with the help of a GC, connected to the reactor chamber as indicated in Figure 2.

A typical propane chromatogram trace is shown in Figure 3(a), as a function of tracing time. The quantity of interest to us is the total counts measured by the GC (red line in Figure 3(a)), which is given by the integral of the excess counts with respect to the reference line without the presence of propane (dashed line in Figure 3(a)).

A set of 16 measurements of the GC area,  $A_{GC}$ , versus the corresponding relative propane flow c is displayed in Figure 3(b). The results suggest a linear regression,

$$A_{GC} = \frac{801 \ c \ (\text{ppm}) + 8.65}{10^4},\tag{2}$$

where  $A_{GC}$  is express in counts and c in ppm. Assuming now that for arbitrary flow conditions we can determine  $A_{GC}$ , we can then estimate the actual propane concentration by inverting Eq. (2), yielding,

$$c \text{ (ppm)} = 10^4 \frac{A_{GC} - 8.65}{801}.$$
 (3)

### **B.** Experimental results

The experimental campaign consists in placing the catalyst and the lamp in the desired position; opening the in-let and out-let valves in order to flow the desired concentration of propane through the reactor for three minutes then closing the valves. A first chromatographic sampling is performed in order to measure the initial propane concentration. When switching on the lamp catalyst starts to act on the propane gas. We analyzed the gas concentrations repeating the sampling at different treatment times. In the reactor box a vent is switched on during the chromatographic sampling in order to flow uniformly the gases towards the chromatograph.

The experimental concentrations are plotted as a function of time in Figures 4-8.

We fit the experimental curves by estimating the propane decomposition simply as

$$\rho(t) = \rho(0) \exp\left(-\frac{t}{\tau}\right),\tag{4}$$

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where  $\rho(0)$  is the initial concentration and  $\tau$  represents the half time depletion. In terms of normalized concentration  $c(t) = \rho(t)/\rho(0)$ , the equation becomes

$$c(t) = \exp\left(-\frac{t}{\tau}\right). \tag{5}$$

We started our experiments using the catalyst A. During this campaign we used different UV-A, UV-C and visible lamps<sup>39</sup>, varying the working conditions such as the propane concentrations and the catalyst-lamp distances.

With the UV-C and visible lamps no propane depletion occurred, while about 7-9% of propane dissociation was detected using UV-A lamp, after 16 minutes of treatment (Figure 4). These results are in agreement with well known photocatalytic activity of  $TiO_2$  in the range of UV-A spectrum.

Tests with UV-A lamp in different positions with respect the catalyst and placing the lamp outside and inside of the reactor box were also performed. The light intensity on the catalyst, when the lamp is outside the box at a distance of 11 cm, is about  $0.4 \text{ mW/cm}^2$ , while when located inside the box at the distance of 0.5 cm, is of about 30 mW/cm<sup>2</sup>. As we can see in Figure 5 propane depletions are similar for the two light intensities and this means that a low intensity is sufficient to ignite the photocatalytic processes.

We performed measurements for different starting propane concentration and we found that the photocatalysis does not depend on the initial concentrations as depicted in Figure 6. Contrary to what happens at low VOC concentrations, where the photocatalytic depletion strongly depends on the concentration itself<sup>40</sup>, at our high initial concentrations, between 1200 and 5000 ppm, the dissociation occurs at similar time rates.

Then we compared the depletion of propane using the three different catalysts A, B and C, and different UV-A, UV-C and visible lamps, at a concentration of about 1200 ppm. Using the UV-A lamp with intensity of 0.4 mW/cm<sup>2</sup>, a propane depletion of 12% occurred with catalyst A, while a dissociation of a few percentages with catalyst B and C was measured (Figure 7). Using UV-C at the intensity of 4 W/cm<sup>2</sup>, only for catalyst B a depletion occurred, of about 5% after 22 minutes. Although the VOC depletion is quite low, this result outlines the photocatalytic activity of the mixture of WO<sub>3</sub> and TiO<sub>2</sub> already reported in literature with UV-C

 $light^{41,42}$ .

To evaluate the ageing of the catalytic support due to exposure to air, we measured the abatement using the catalyst A deposited on the tile support since two months. In this experiment a lower depletion trend was observed, as reported in Figure 8. This result permits to estimate the time life of our catalytic support.

### IV. CONCLUSIONS

In this paper we demonstrated the ability of  $TiO_2$  catalysts to abate high propane concentrations using relatively low intensity UVA lamp, of about 0.4 mW/cm<sup>2</sup>.

We performed an experimental reactor in order to study the propane abatement at high concentrations by catalytic processes using TiO<sub>2</sub>, WO<sub>3</sub> and their mixtures deposited on a tile which surface is about 55 cm<sup>2</sup>, and UV-A, UV-C and visible lights. The highest abatement has been obtained using TiO<sub>2</sub> catalyst and UV-A light. The abatement rate for initial propane concentration between 1200 and 5000 ppm is of the order of 10% after 22 minutes and the time scale of depletion is of the order of hours. We found that the TiO<sub>2</sub> photocatalytic activity does not depend on the initial concentration contrary to what happens at low VOC concentrations, as reported in literature<sup>40</sup>.

The TiO<sub>2</sub> catalyst suffers of ageing effects, since after two months its depletion action is greatly reduced. A relatively lower abatement is detected by UV-C lamp when TiO<sub>2</sub> and WO<sub>3</sub> mixture is used, of about 5% in 22 minutes. Null abatement was measured when using visible lamps for all the catalysts, and when using WO<sub>3</sub>.

This analysis demonstrates the efficacy of the photocatalysis for high VOC concentrations, and also represent a starting point for our further studies on the hybrid plasma-catalysis were  $\text{TiO}_2$  catalyst will be deposited in vicinity of the plasma electrodes.

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and Alessandro Baù in the device development and experiment execution.

### AVAILABILITY OF DATA

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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FIG. 1: Reactor setup (a) scheme and (b) photo. The lamp can be placed both inside or outside the reactor box. The reactor is equipped with a quartz window to let the lamp light from the outside activate the catalyst, a vent to recirculate the air, a catalyst, a gas chromatograph inlet and air inlet and outlet valves.

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FIG. 2: Equipment setup. The VOC gas (propane) is placed in a cylinder and its flow is regulated by the flow meter 1, while the pure air cylinder flow is regulated by the flow meter 2. The analyses are made using a Micro GC Agilent 3000 gas chromatograph.

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FIG. 3: (a) Example of propane chromatogram peak and (b) the calibration plot of propane reporting 16 couples of data (AreaGC and c) measured varying the propane in-let in the reactor box.



FIG. 4: Propane depletion using UV-A at the intensity of 30 mW/cm<sup>2</sup>. About 10% of pentane was abated. Both graphs are in y-log scale, (a) in the y range [0.6-1.02], while (b) in [0.88-1.02].



FIG. 5: Propane depletion using UV-A at different distance between lamp and catalyst, that means a different intensity of the lamp.

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FIG. 6: Propane depletion using UV-A and different starting propane concentration.



FIG. 7: Propane depletion using catalist A, B and C using UV-A lamp.

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FIG. 8: Propane depletion using catalist A and UV-A lamp for different time after catalyst deposition.



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