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Using in situ measurements to experimentally characterize TiO₂ nanoparticle synthesis in a turbulent isopropyl alcohol flame

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Abstract: The objective of the present work is to show the potential of in situ measurements for the investigation of nanoparticles production in turbulent spray flames. This is achieved by considering multiple diagnostics to characterize the liquid break-up, the reactive flow and the particles production in a spray burner for TiO₂ nanoparticle synthesis. The considered liquid fuel is a solution of isopropyl alcohol and titanium tetraisopropoxide (TTIP) precursor. Measurements show that shadowgraphy can be used to simultaneously localize spray and nanoparticles, light scattering allow to characterize the TiO₂ nanoparticles distribution in the flame central plane, and spontaneous CH* and OH* chemiluminescences as well as global light emission results can be used to visualize the reactive flow patterns that may differ with and without injection of TTIP. Concerning the liquid, it is observed that is localized in a small region close to the injector nozzle where it is dispersed by the oxygen flow resulting in droplets. The liquid droplets rapidly evaporate and TTIP is quasi-immediately converted to TiO₂ nanoparticles. Finally, results show high interactions between nanoparticles and the turbulent eddies.

Keywords: In situ optical diagnostics; flame synthesis; TiO₂; turbulence

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1. Introduction

Synthesis in turbulent spray flames is today considered as a valuable alternative for large-scale production of nanoparticles with a relatively low cost. Laboratory-scale spray flame reactors [1–12] were therefore developed to improve our understanding of nanoparticles production in these reactive flows in order to better control the characteristics and properties of the final product. For this, ex situ measurements are classically performed to characterize the collected materials in terms of morphology, physical and optical properties depending, for example, on operating conditions such as temperature, pressure, and precursor concentration. However, it is expected that the properties of nanoparticles produced via flame synthesis will depend on the experienced local conditions governed by the flow and the flame. Therefore, it would be of interest to combine classical ex situ measurements to in situ optical diagnostics classically used in combustion research to understand the physical processes occurring during the flame synthesis by characterizing the spatial and temporal evolution of spray, flow, flame and nanoparticles. In addition, in situ measurements will allow the characterization of boundary conditions necessary to perform numerical simulations [13–17] and they provide an experimental database for their validation [9,18].

In this framework, the present study aims to prove the feasibility and the great interest of in situ measurements when investigating nanoparticles production in a laboratory-scale spray flame reactor. The burner consists of a spray nozzle where the liquid fuel is atomized by an annular flow of oxygen, a circular pilot premixed ethylene/air flame and a coflow of pure N₂. The considered liquid fuel is a solution of isopropyl alcohol and titanium tetraisopropoxide (TTIP) precursor.

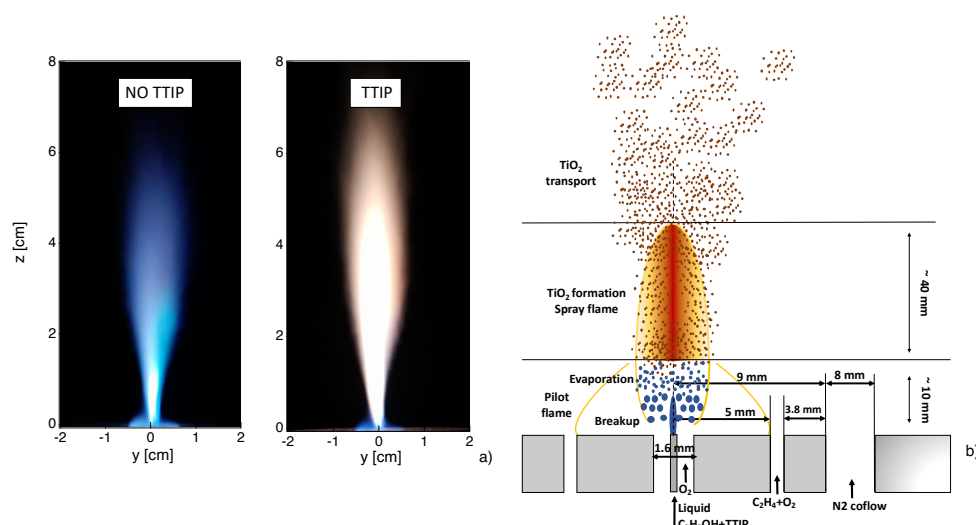


Figure 1. a) Flame luminosity without and with injection of TTIP. b) Schematic presentations of the reactor inlet and of the processes inside the reactor.

38 Three main processes characterize the flame: a) the break-up of the liquid jet, b) the
 39 turbulent reactive flow, and c) the production of the particles. The spatial localization
 40 of these three processes is here provided using in situ experimental measurements,
 41 classically used in combustion research: a) shadowgraphy and light scattering for the
 42 liquid phase, b) flame luminosity, CH^* and OH^* chemiluminescences to characterize the
 43 combustion process, and c) light scattering to localize TiO_2 nanoparticles.

44 The paper is organized as follows. First, the experimental setup is described by
 45 presenting the flame synthesis burner and the different optical diagnostics considered
 46 in this work. Then, the potentials and the difficulties in applying shadowgraphy and
 47 light scattering to the characterization of nanoparticles flame synthesis are discussed.
 48 Finally, results are presented by looking at the three different processes that characterize
 49 nanoparticles flame synthesis.

50 2. Experimental setup

51 2.1. Flame synthesis burner

52 The burner studied in the present work is the same as used in [2–5,15], the ParteQ
 53 GMBH model LS-FSR. The burner, schematically presented in Fig. 1b, allows the sta-
 54 bilisation of a turbulent spray flame, whose luminosity is visualized in Fig. 1a. For
 55 this, liquid isopropyl alcohol (Sigma Aldrich, C_3H_8O) is injected through a syringe in
 56 the center. The liquid flow is provided by a Tuthill pump upstream of a mini-Coriolis
 57 flowmeter from Bronkhorst. The imposed flow rate is 0.005 Nl/min. The liquid jet is
 58 surrounded and dispersed by an annular jet of pure oxygen with a flowrate of 3 Nl/min.
 59 A premixed methane-oxygen pilot flame with an equivalence ratio of $\Phi = 0.83$ (oxygen
 60 flowrate of 1.2 Nl/min and methane flowrate of 0.5 Nl/min) is needed to stabilize the
 61 non-premixed flame. The coflow consists of pure nitrogen with a flowrate of 4 Nl/min.

62 The obtained flame is visualized in Fig. 1a. As already observed in [15], the flame is
 63 not perfectly axis-symmetric mainly due to a non-symmetric pilot flame as a result of
 64 some geometrical imperfections of the burner. To consider TiO_2 nanoparticles production,
 65 titanium tetraisopropoxide (TTIP), $Ti(OCH(CH_3)_2)_4$, (Sigma Aldrich) with a purity of
 66 97% is added to liquid flow in a proportion of 5 ml of TTIP for one litre of isopropyl
 67 alcohol. The obtained flame is visualized in Fig. 1a. It can be observed that a more
 68 luminous flame is obtained when considering injection of TTIP.

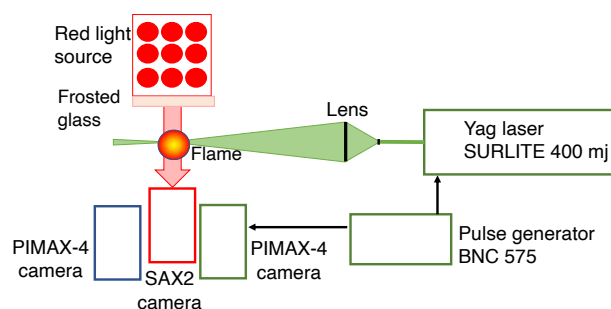


Figure 2. Schematic presentation of the optical setup.

69 2.2. *In situ* optical diagnostics

70 The optical setup used to perform shadowgraphy, light scattering and emission
 71 measurements is schematically presented in Fig. 2. The shadowgraph measurements
 72 are performed using a red backlighting system featuring a red LED spot. The red LED
 73 spot consists in a 7 cm x 9 cm rectangle of LEDs emitting at a dominant wavelength
 74 of 633 nm. A frosted glass is placed between the spot and the flame to get a light as
 75 homogeneous in space as possible. The spot is fed by a direct current power supply in
 76 order to avoid main current frequency interference. A Photron Fastcam SAX2 camera is
 77 placed at the opposite side of the LED spot to obtain shadowgraphy images of the flame.
 78 It is equipped with a Nikon 105mm f/2.8 lens and a 20 mm extension ring. Images of
 79 1024x1024 pixels are obtained with a resolution of 62 $\mu\text{m}/\text{pixel}$. The signal acquisition
 80 gate width is 100 μs .

81 A YAG Surelite 400 mJ Continuum laser at 532 nm wavelength is used for the light
 82 scattering on the solid particles. A set of two lenses creates a laser sheet of 70 mm height
 83 and about 300 μm thickness, which passes through the burner central axis. The scattered
 84 light is captured by a Teledyne Princeton PIMAX4 camera (1024 * 1024 pixels) equipped
 85 with a Nikon lens 100F/1.8 and a 20 mm extension ring. It has a spatial resolution of 62
 86 $\mu\text{m}/\text{pixel}$. A Semrock FF01 530nm FWHM 11 nm filter allows the observation of only
 87 light scattering. Both the camera and the laser are synchronized via a pulse generator
 88 BNC575. The images are captured with no delay and a gate width of 15 ns.

89 A second Teledyne Princeton PIMAX camera equipped with a Soder UV lens
 90 100F/2.8 is used for measurements of flame global spontaneous emission as well as CH*
 91 and OH* emissions. The spatial resolution is 95 $\mu\text{m}/\text{pixel}$. For flame global spontaneous
 92 emission, no filter is used on the camera and the exposure time is adjusted in order to not
 93 saturate the gray levels (5 μs). Regarding the OH* spontaneous emission, an Asahi 310
 94 nm filter (96SA02), FWHM 10.00 nm is used in front of the camera lens. The exposure
 95 time is 30 μs . For CH* spontaneous emission, an Asahi 430 nm filter (F0102), FWHM
 96 10.00 nm filter is used and an exposure time of 15 μs is retained.

97 Time-averaged results for all measurements are obtained by subtracting the back-
 98 ground and considering 500 images.

99 3. Using shadowgraphy and light scattering diagnostics to characterize flame 100 synthesis

101 Several phenomena can be visualized using the shadowgraphy measurements as
 102 illustrated in Fig. 3a. First of all, when considering the non-reacting cold case, the
 103 presence of the liquid jet and of the spray can be detected since the objects between the
 104 light source and the camera appear the darker the more they absorb the light. Similarly,
 105 the presence of liquid jet is detected in both reacting cases without and with injection of
 106 TTIP. It can be observed that the liquid phase occupies a smaller region, compared to the
 107 cold case, due to its quick evaporation due to high flame temperature.

108 In the reactive case, with the injection of TTIP, spots of light due to diffraction of
 109 partially transparent TiO_2 nanoparticles can also be observed. Then, it is possible to

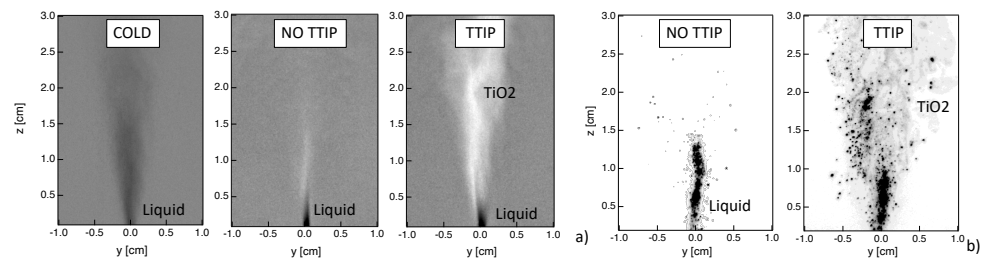


Figure 3. Instantaneous images obtained by (a) shadowgraphy and (b) light scattering, without and with injection of TTIP. Results for the non-reacting cold case with shadowgraphy are also presented.

110 discriminate spray from TiO_2 nanoparticles by considering dark or bright information.
 111 Therefore, thanks to the shadowgraphy, it is possible to get simultaneous information on
 112 the localization of both spray and of TiO_2 nanoparticles.

113 Since the shadowgraphy measurements provide line-of-sight-integrated informa-
 114 tion, light scattering measurements were also performed to investigate spray and TiO_2
 115 nanoparticles distribution at the burner central plane. Instantaneous images for both
 116 cases with and without injection of TTIP are presented in Fig. 3b. Although planar
 117 information can be obtained with this technique, however, a rigorous discrimination
 118 between the signals from spray and TiO_2 nanoparticles is not straightforward. It has
 119 been observed that, in the region close to the liquid injection, the contribution from the
 120 liquid spray scattering is predominant compared to the one from nanoparticles. There-
 121 fore, it is assumed in the following that, in the spray zone close to the burner, a high
 122 intensity signal corresponds to spray light scattering, whereas low intensity corresponds
 123 to TiO_2 nanoparticles. Even if such criterion is arbitrary, the complementary use of
 124 shadowgraphy allows to identify the region where the liquid phase is expected to be
 125 observed (in our case for a height above the burner $z < 2$ cm). Indeed in this region,
 126 results from light scattering should be analyzed with caution, but beyond this region,
 127 light scattering information can be used to localize the presence of TiO_2 particles.

128 4. Characterization of spray flame synthesis

129 The main processes occurring during the flame synthesis, schematically presented
 130 in Fig. 1b, are described here thanks to in situ optical diagnostics, classically used in
 131 combustion research, comparing results on flames with and without injection of TTIP.

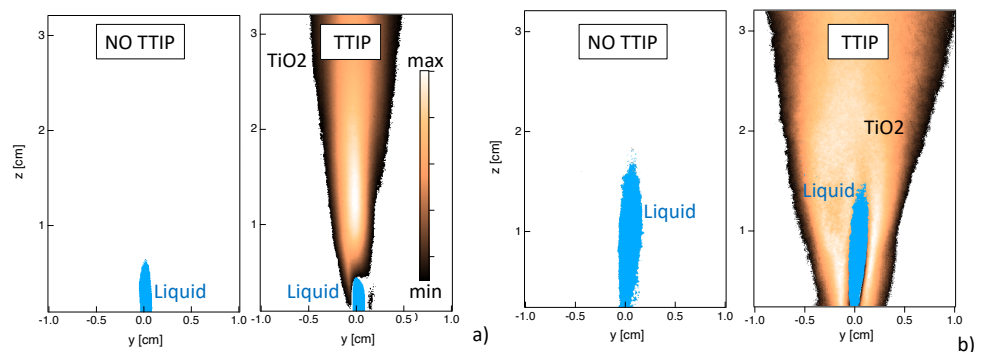


Figure 4. Localization of liquid phase (blue color) and TiO_2 nanoparticles (brown palette) via (a) shadowgraphy, and (b) light scattering. Time-averaged results (left) without and (right) with injection of TTIP are presented.

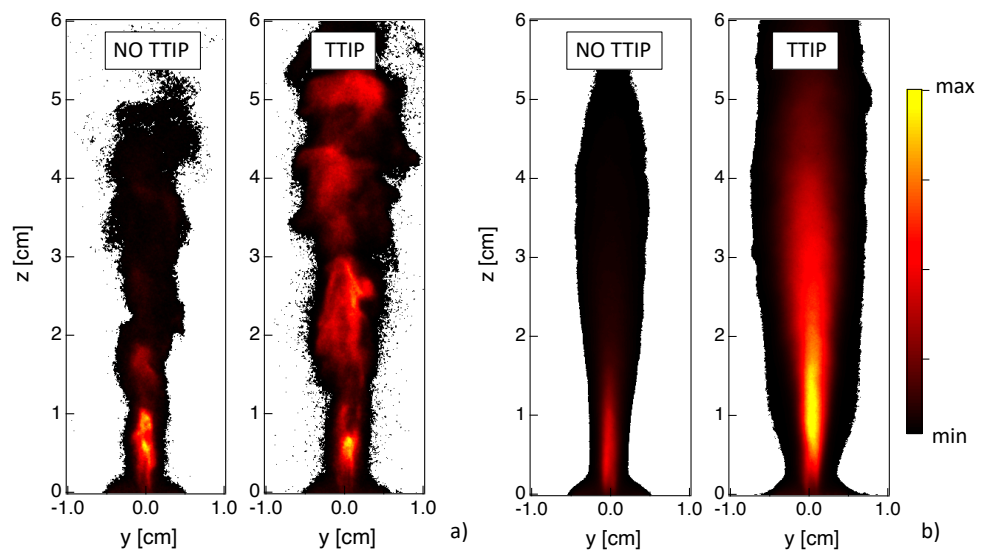


Figure 5. (a) Instantaneous and (b) time-averaged images of line-of-sight-integrated global emission for the reactive cases (left) without and (right) with injection of TTIP.

132 4.1. Liquid injection and spray

133 Thanks to shadowgraphy and light scattering measurements, the presence of liquid
 134 can be investigated. When looking at the instantaneous results of Fig. 3, the presence
 135 of a central liquid jet core is observed. The liquid is localized in a small region close
 136 to the injector nozzle due to the effect of the dispersion oxygen flow, which leads to
 137 the break up of the liquid jet into droplets, together with the effect of the flame high
 138 temperature, which results in a rapid evaporation. Occasionally, big droplets can be
 139 observed at higher heights above the burner.

140 It should be noted that some differences are observed between the two techniques.
 141 In particular, a dense cylindrical liquid jet seems to be detected by light scattering
 142 whereas the atomization seems to occur more rapidly from shadowgraphy results.
 143 However, it has to be reminded that the two systems neither present the same sensitivity
 144 nor the same resolution of the liquid structures. Moreover, line-of-sight-integrated
 145 measurements are provided by shadowgraphy, whereas light scattering gives access to
 146 planar information. Finally, light scattering results may be affected by the fact that both
 147 spray and TiO_2 nanoparticles are simultaneously detected.

148 Time-averaged shadowgraphy and light scattering results are presented in Fig.
 149 4 for the flames without and with injection of TTIP. A slightly shorter spray region is
 150 identified in the case of the flame with TTIP. However, no other significant differences are
 151 observed so that it can be deduced that the spray region is correctly identified by the light
 152 scattering technique even in the presence of nanoparticles. Therefore, the differences
 153 between results from light scattering and shadowgraphy are most probably due to the
 154 intrinsic specificity of these two techniques.

155 Even if a more detailed characterization of the performances of these techniques
 156 in the context of flame synthesis is desirable, some common conclusions on the spray
 157 process can already be drawn. First, high fluctuations of the spray position are observed
 158 (not shown). Second, results are not symmetric, possibly due to the difficulty in obtaining
 159 a perfect centering of the liquid injection syringe in the dispersion system. Third, TiO_2
 160 nanoparticles are formed close the spray, indicating that the nanoparticles production is
 161 an extremely fast process occurring once the TTIP precursor has evaporated. Finally, it
 162 can be said that the spray is not likely to be found for $z > 2$ cm, so that in this region light
 163 scattering from spray can be considered as negligible compared to nanoparticles contri-
 164 bution. Therefore, results on the localization of TiO_2 nanoparticles can be considered
 165 with confidence for $z > 2$ cm.

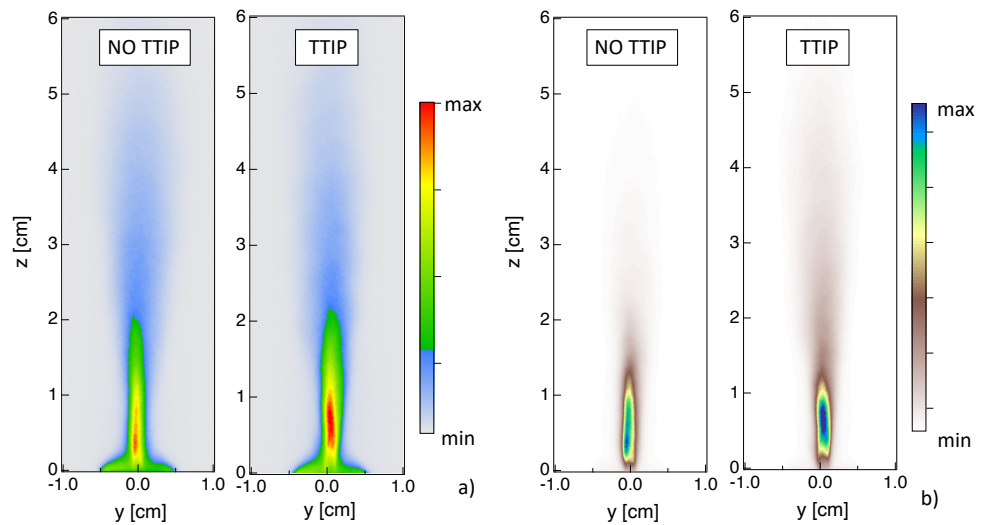


Figure 6. Time-averaged line-of-sight-integrated of (a) OH^* and (b) CH^* emission for the reactive cases (left) without and (right) with injection of TTIP.

166 4.2. Flame

167 The combustion process is investigated here by analyzing images of flame global
 168 spontaneous emission (Fig. 5) as well as CH^* and OH^* chemiluminescences (Fig. 7).
 169 The global spontaneous emission from the flame contains information on the whole
 170 flame emission, while CH^* and OH^* chemiluminescences can be used to localize the
 171 heat release zone. In the presented case, the signal from global spontaneous emission is
 172 generally 10 times more intense than the one from OH^* and CH^* .

173 Looking at the instantaneous global emission results, Fig. 5a, a turbulent flame
 174 structure can be recognized even if these measurements provide line-of-sight-integrated
 175 information. When considering time-averaged results, Fig. 5b, it can be noticed that the
 176 symmetry of the fields is not perfect, similarly to the results for spray in Fig. 3.

177 Results are quite different between the two cases with and without injection of TTIP,
 178 indicating that the addition of TTIP has a non-negligible effect on global spontaneous
 179 emission, as already deduced from Fig. 1a. When looking at the case without TTIP, the
 180 most relevant emission contribution due to the isopropyl flame is located along the
 181 central line at small height above the burner ($z < 1$ cm). The pilot flame is identified by
 182 the small conical emission region localized close to the burner tip and it only slightly
 183 contributes to the flame emission. In the case with injection of TTIP (Fig. 5b), the
 184 maximum values of emission are found far above the burner ($0.5 \text{ cm} < z < 1.5 \text{ cm}$) due
 185 to the presence of nanoparticles. In this case, spontaneous emission is the result of both
 186 flame and nanoparticles emissions. The maximum values of spontaneous emission for
 187 the case with injection of TTIP are higher by a factor of 10, compared to the case without
 188 injection of TTIP.

189 Time-averaged fields of OH^* and CH^* chemiluminescences are presented in Fig. 6.
 190 Concerning results without injection of TTIP, CH^* and OH^* chemiluminescence signals
 191 present a similar spatial evolution compared to global emission. Most of the heat is
 192 expected to be released close to the region where spray evaporation occurs ($z < 2$ cm),
 193 with a maximum located at $z < 1$ cm. Post-combustion processes are observed up to
 194 $z \approx 5$ cm. Close to the injector, a lower signal intensity is measured compared to the
 195 central region, possibly indicating that the pilot flame only slightly contributes to the
 196 global heat release even if it is essential for the flame stabilization. Results with injection
 197 of TTIP are qualitatively similar to those without TTIP, even if more intense signals are
 198 observed for the TTIP case. This is probably due to the fact that the addition of TTIP
 199 leads to an increase of carbon atoms compared to a pure isopropyl alcohol flame. By
 200 comparing these results with those on global emission for the TTIP case in Fig. 5, it can

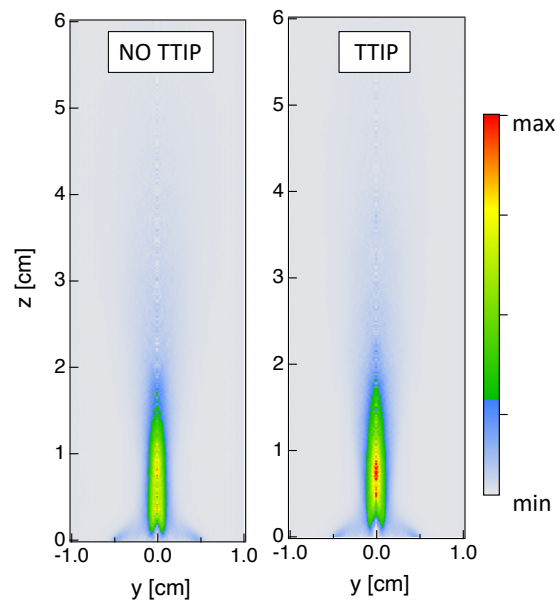


Figure 7. Planar information are extracted using the Abel-inversion on time-averaged results from OH* chemiluminescence for the reactive cases (left) without and (right) with injection of TTIP.

201 be confirmed that the maximum of global emission at $z > 1$ cm is due to the presence of
 202 particles.

203 Classically, time-averaged line-of-sight integrated results could be transformed
 204 using an Abel-inversion to obtain 2-D planar information. Since the investigated flame
 205 is not quite axis-symmetric, as can be deduced by looking for example at the CH* results
 206 in Fig. 6b), caution should be paid when analysis Abel-inverted results. An example
 207 of planar results is presented in Fig. 7 considering only the right half-side of the time-
 208 averaged results from OH* chemiluminescence. When looking at 2-D planar fields, a
 209 lower signal intensity is measured close to the injector compared to the central region.
 210 Therefore, it is quite evident by looking at 2-D fields that the pilot flame only slightly
 211 contributes to the global heat release even if it is essential for the flame stabilization.
 212 Globally, similar conclusions can be deduced compared to line-of-sight integrated results
 213 in terms of localization of maximum value of OH* signal and effect of TTIP addition on
 214 OH* emission.

215 4.3. Particles production

216 Figure 8 presents light scattering measurements of TiO₂ nanoparticles by gathering
 217 random collections of data at different vertical positions above the burner. As mentioned
 218 above, the presence of TiO₂ nanoparticles can be analyzed by looking at both shadowg-
 219 raphy and light scattering fields. When looking to results close to the injector (Fig. 4), it
 220 can be seen that nanoparticles appear in the close proximity of the spray. This indicates
 221 that, once the liquid TTIP precursor has evaporated, it is rapidly converted into solid
 222 TiO₂ particles, confirming that TiO₂ production is governed by fast reactions. Once the
 223 particles formed, their localization seems to be strongly governed by turbulent eddies, as
 224 it can be observed from the instantaneous results of light scattering close to the injector
 225 (Fig. 4b) and along the flame (Fig. 8a). Then, TiO₂ particles mainly concentrate along
 226 thin ligaments that are stretched and deformed by the turbulent flow eddies and are
 227 finally convected downstream the combustion region ($z > 4$ cm).

228 Far downstream of the burner ($z > 7$ cm), a more homogeneous spatial distribution
 229 of TiO₂ particles is observed due to turbulent mixing. Time-averaged results of light
 230 scattering are presented in Fig. 8b. A very intense signal is observed close to the burner,
 231 which decreases downstream the post-flame region. This high light scattering region
 232 seems to coincide with the high flame luminosity zone in Fig. 1a. The light scattering

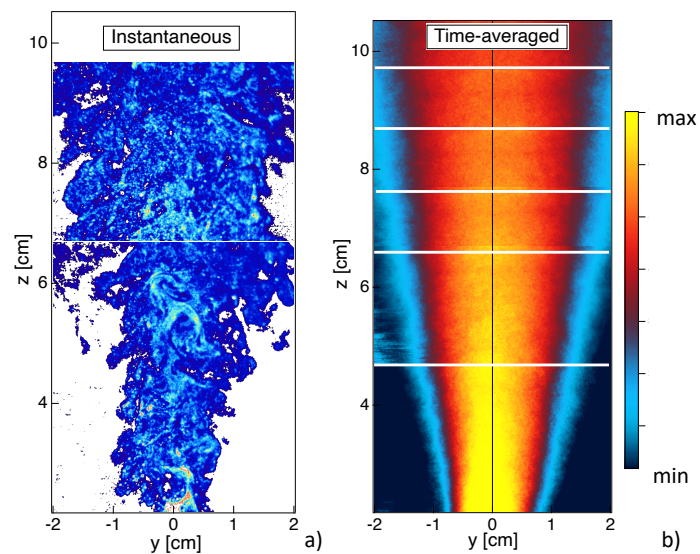


Figure 8. Localization of TiO_2 nanoparticles via light scattering measurements. Instantaneous (left) and time-averaged (right) fields. Time-averaged field is reconstructed by assembling series of measurements at six vertical positions of the laser sheet.

233 signal depends on particle size and number density. Since the size of the particles is not
 234 expected to decrease along the height above the burner, the light scattering field seems to
 235 indicate that the nanoparticles are generated close the spray region in large number and
 236 that the particles number density subsequently decreases due to collisional processes.
 237 Even if the distribution of the light scattering signal is not symmetric close to the burner
 238 (Fig. 4b) due a non axis-symmetric flame, its time-averaged distribution becomes rapidly
 239 symmetric, probably due to the turbulent transport of the particles downstream.

240 5. Conclusion

241 The objective of the present work was to demonstrate the interest of in situ opti-
 242 cal diagnostics classically used in combustion research for the investigation of TiO_2
 243 nanoparticles synthesis in turbulent spray flames. Shadowgraphy, light scattering, and
 244 global flame luminosity as well as CH^* and OH^* chemiluminescence measurements
 245 were employed in order to study the three main processes that characterize the spray
 246 flame. In this way, the liquid break-up, the reactive flow and the TiO_2 nanoparticles
 247 production were analyzed.

248 Shadowgraphy measurements showed that it was possible to simultaneously local-
 249 ize the liquid phase and the nanoparticles. Light scattering results allowed to character-
 250 ize the TiO_2 nanoparticles distribution in the flame central plane. The liquid flow is localized
 251 in a small region close to the injector nozzle where it is dispersed by the oxygen flow
 252 resulting in droplets. The liquid droplets rapidly evaporate due to high temperature
 253 of the flame. When TTIP is added to the liquid flow, right after its evaporation and
 254 due to its high reactivity, it is immediately converted to TiO_2 nanoparticles. Global
 255 spontaneous emission is quite different when considering TTIP compared to the flame
 256 without TTIP. In specific, when TTIP is added, maximum emissions are observed far
 257 above the burner showing the non-negligible contribution of TiO_2 particles emissions.
 258 On the contrary, even if CH^* and OH^* chemiluminescence signals are more intense when
 259 adding TTIP, the signals are qualitatively in agreement with the flame without TTIP.
 260 Finally shadowgraphy and light scattering results at different heights above the burner
 261 showed high interactions between nanoparticles and the turbulent eddies. Even if in
 262 future works an optimization of these techniques to flame synthesis is desirable, in situ
 263 optical diagnostics from combustion research can be used to provide a new insight on
 264 flame synthesis, complementary to ex situ measurements.

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