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Additional Information

1 **Application of optical diagnostics to the quantification of soot in**
2 **n-alkane flames under diesel conditions**

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16

17 **Abstract**

18 In the present paper, three different soot-measuring techniques, namely Laser Extinction
19 Method (LEM), 2-Color Pyrometry (2C) and Laser-Induced Incandescence (LII) have been
20 simultaneously employed to characterize soot distribution inside a diesel flame. Two
21 single-component fuels (n-Decane and n-Hexadecane) and two derived blends
22 (50%Dec/50%Hex and 30%Dec/70%Hex) have been used. Tests have been performed at an
23 optical diesel engine, under different in-cylinder conditions. The study has been complemented
24 with the measurement of ignition delay and Lift-off length.

25 The present work pursues a twofold objective. On the one hand, the effect of fuel properties on
26 soot formation have been analysed, under different engine operating conditions. On the other
27 hand, sensitivity and performance of the three optical techniques has been evaluated,
28 identifying their main advantages and drawbacks in the framework of the current study. LEM
29 has been considered as the reference technique, as the measurement principle can be
30 implemented without important limitations associated to the other two. Results highlight that
31 larger molecules produce more soot than the smaller ones, with both reactivity and soot
32 formation changing with the proportion of the heavier fraction. Despite describing similar
33 trends, LEM and 2C do not provide the same KL values, with the pyrometry reaching some sort
34 of saturation when increasing flame soot. A detailed analysis confirms that 2-Color
35 measurements are strongly biased by soot and temperature distribution inside the flame.
36 Nevertheless, it could still be a good option for low sooting conditions. On the other hand, an
37 attempt to calibrate LII signal by means of LEM measurements has been reported. This approach
38 should make it possible to obtain additional information on the soot spatial distribution.
39 However, inconsistencies have been identified which stem from the inherent limitations of LII
40 technique in highly sooting conditions.

41 **Keywords:**

42 Soot Measurement, Laser induced incandescence, two-colour pyrometry, laser extinction
43 method, n-alkane, Diesel combustion

44 **1. INTRODUCTION**

45 Optical diagnostic techniques have been traditionally used to improve the insight on the basic
46 phenomena that dominate combustion within internal combustion engines. In particular, the
47 characterization of the soot formation during combustion is a challenging topic, as it involves

48 complex physical and chemical processes that dominate both formation and later oxidation [1,
49 2]. Three main optical techniques can be found in the literature for the study of this topic: Laser-
50 Induced Incandescence (LII), 2-Colour Pyrometry (2C) and Laser Extinction Method (LEM). They
51 have been applied traditionally to diesel flames, but they present certain advantages and
52 drawbacks that must be considered before choosing the most suitable tool for each specific
53 study.

54 LII is based on recording the high intensity radiation emitted by soot particles that are previously
55 heated by a laser pulse. The magnitude of the signal can be correlated with the volume fraction
56 of particles in the detection region. This technique has been used extensively for qualitative [3–
57 6] and even quantitative measurements [7-9]. However, quantitative measurements require a
58 firm understanding of the factors that influence the LII signal, which are detailed in [10]. As an
59 intermediate step, calibration of LII signal with LEM measurements is quite often employed [11–
60 16], in theory should make it possible to derive 2D soot volume fraction distributions. This
61 approach, however, does not solve the inherent limitations of LII related to radiation
62 attenuation processes within the flame.

63 2-Colour Pyrometry is based on the detection of the spontaneous thermal radiation emitted by
64 incandescent soot particles at two different wavelengths. The technique makes it possible to
65 obtain not only the soot distribution but also the corresponding temperature. Moreover,
66 modern high-speed cameras offer a high time resolution. This technique has been widely used
67 by the diesel engine research community [17-27]. However, the analysis of results does not
68 always consider the intrinsic limitations of the technique [22, 23].

69 While 2C and LII could be strongly affected by the interaction of emitted radiation with other
70 soot particles within the flame, the third technique, LEM, is just based on this property. The
71 attenuation can be related to the optical thickness of the soot cloud and, eventually, to the soot
72 volume fraction. This technique has been widely used in single diffusion flames [28-31] and, with
73 the proper considerations [30], reliable results can be obtained. LEM applications are based on
74 point measurements along the flame, using a small laser beam. It allows high time resolution
75 but it is spatially limited by the beam size. However, nowadays applications start to appear
76 where a larger light source is combined with high-speed cameras, offering both good spatial and
77 time resolution [32].

78 In the present work, the three techniques (LII, 2C and LEM) have been proposed to characterize
79 soot formation under diesel engine conditions. They have been applied simultaneously, to
80 evaluate the effect of physical and chemical properties of two single-component surrogates

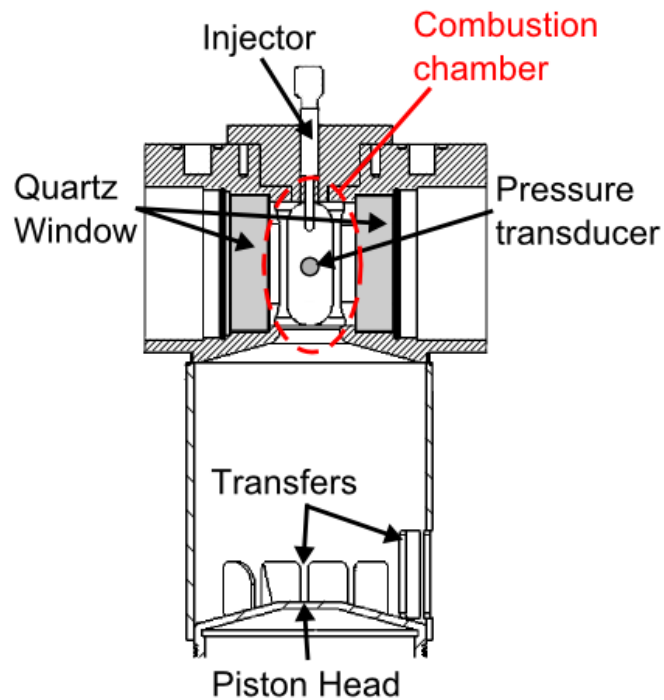
81 (n-Decane and n-Hexadecane) and two derived blends. The analysis of experimental results will
82 make it possible to fulfil a twofold objective: firstly, describe the effect of fuel properties over
83 soot formation; secondly, identify the strengths and limitations of each methodology. The first
84 part of the paper presents a detailed description of the experimental apparatus and procedure.
85 Then, a comparison among results obtained by each technique is presented. Trends and
86 numerical results are analysed and discussed, trying to clarify the main differences observed.
87 Finally, the main conclusions regarding the influence of fuel properties and the optical
88 techniques, together with recommendations for the proper use of these experimental tools, will
89 be summarized.

90 **2. EXPERIMENTAL METHODOLOGY**

91 **2.1. Experimental Test Bench**

92 All the tests have been performed at an optically accessible single cylinder engine, which is
93 described in detail in [33]. The facility is based on a 2-stroke single cylinder direct injection diesel
94 engine (Jenbach JW 50), with three liter displacement and 15.7 effective compression ratio. It is
95 motored at low engine speed (500 rpm). Intake and exhaust processes are handled by transfers
96 on the liner and the cylinder head is specially designed to provide optical access to the
97 combustion chamber. A cylindrical combustion chamber was designed in a way that spray wall
98 impingement is avoided. The chamber has an upper port where the injector can be mounted
99 and four lateral accesses. A pressure transducer is installed in one of the accesses, whereas the
100 other three are equipped with oval-shaped quartz windows, 88 mm long, 37 mm large and 28
101 mm thick. A cutaway view of the cylinder head is depicted in Figure 1. The cylinder head and the
102 engine temperature are controlled by means of coolant recirculation. Their temperature was set
103 to 353 K, to guarantee a good performance of the lubricant oil.

104 In-cylinder thermodynamic conditions during the cycle are controlled by the intake air
105 temperature and pressure. The first one is regulated by two sets of electrical resistors, while the
106 desired intake pressure is achieved thanks to a compressor that is fed with ambient air. The
107 engine is operated under skip-fired mode, so that in-cylinder conditions are not influenced by
108 the remaining residual gases from previous combustion cycles. An injection takes place each 30
109 cycles, which guarantees that ambient conditions are kept constant between consecutive
110 repetitions and temperature transients are avoided.



111

112

Figure 1 Cutaway view of the cylinder head layout

113

A common-rail injection system is used, together with a single-hole piezoelectric injector with a 140 μm outlet diameter nozzle. The injector hole is 1 mm long with conical shape (Ks factor of 1.5). The injected mass is so low that thermodynamic conditions inside the combustion chamber are barely affected by the fuel evaporation [24]. Due to the low injection frequency used during tests, the injected fuel initial temperature can be considered the same.

116

118 2.2. Experimental procedure

119

Two single-component fuels have been used, namely n-Decane and n-Hexadecane, together with two derived binary blends: 50%Decane/50%Hexadecane and 30%Decane/70%Hexadecane (percentages in mass). The main advantage of using such simple fuels is that it is expected that they will form less soot than other fuels like commercial diesel, thanks to the absence of ring or branched structures as well as sulphur [2]. The most relevant properties of the fuels for the purposes of this work are given in *Table 1*.

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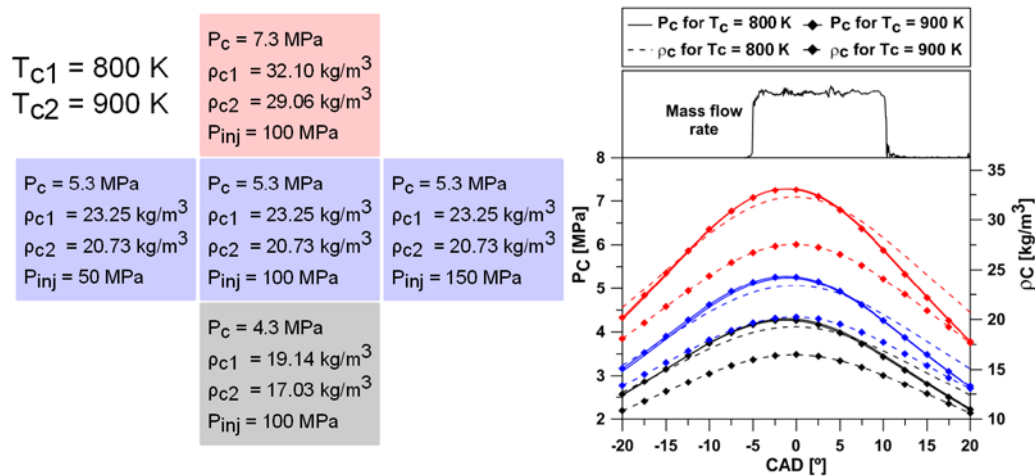
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Fuel	Density at 373 K [Kg/m ³]	Formula	Derived Cetane number	C-C Bonds	H/C
n-Decane	669.2	C ₁₀ H ₂₂	65.9	9	2.19
50Dec/50Hex	693.9	-	82.2	11.37	2.16
30Dec/70Hex	703.7	-	85.4	12.56	2.14
n-Hexadecane	718.5	C ₁₆ H ₃₄	92.9	15	2.12

128

Table 1 Fuel properties.

129 The full test matrix comprises a combination of two in-cylinder top dead center (TDC)
 130 temperature values (800/900 K) with three different TDC pressure values (4.3, 5.3 and 7.3 MPa)
 131 and three injection pressures (50/100/150 MPa). In-cylinder thermodynamic conditions (Figure
 132 2 -right-) have been calculated from measured in-cylinder pressure, using a first-law
 133 thermodynamic analysis as it can be found in [31, 33]. The model takes into account blow-by,
 134 heat losses and mechanical deformations. The trapped mass is estimated using the intake
 135 temperature and volume at the exhaust vent close. Then, temperature along the engine cycle
 136 can be calculated using the equation of state and correcting the trapped mass with blow-by
 137 estimations.



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139 Figure 2 Test matrix (left) and the corresponding thermodynamic conditions in the combustion chamber

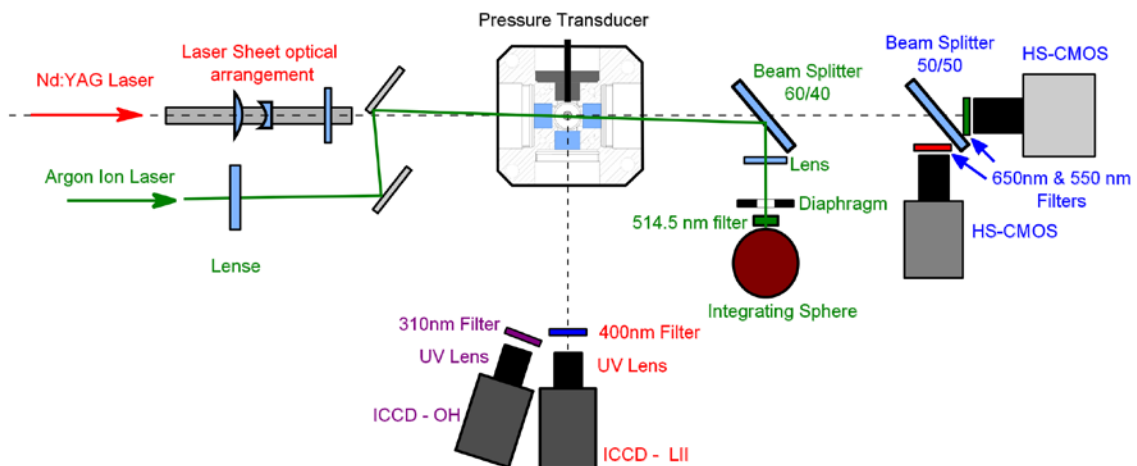
140 As previously mentioned, three different optical techniques have been applied simultaneously
 141 to measure soot formation inside the flame. The injector energizing time was set to 3 ms (9 CAD)
 142 for all conditions, which results in an approximate 6 ms (18 CAD) real injection duration,
 143 considering electrical and hydraulic delays. The injector was triggered at 6 CAD before TDC (SoE),
 144 while the injection started at approximately 5 CAD before TDC (SoI), so that the variations of the
 145 in-cylinder thermodynamic conditions during the injection event were minimized. The 2-Color
 146 Pyrometry and Laser Extinction Method are able to measure the soot formation during the
 147 whole combustion event, thanks to the high sampling rate of the detectors. On the one hand,

148 two high-speed cameras (2C) were set to start registering the light emitted by the flame at SoE,
 149 with $\Delta t = 66 \mu s$ (0.2 CAD) between two consecutive frames. On the other hand, a fast-response
 150 photodiode (LEM) was continuously measuring the intensity of a laser that was aligned
 151 perpendicular to, and intersecting, the flame axis. Finally, for each injection event a Nd:YAG laser
 152 was fired at 3 CAD after SoE, to measure the induced incandescence from soot. This instant was
 153 chosen in order to guarantee the stabilization of the diffusion flame before the laser was fired.

154 2.3. Optical System

155 Different optical techniques have been applied simultaneously, taking advantage of the three
 156 available optical accesses located in the cylinder head. The optical arrangement is shown in
 157 Figure 3. In the following subsections, a more detailed description on the soot techniques is
 158 presented. Additionally, images of OH* radiation with an ICCD camera were recorded
 159 simultaneously with the soot measurements. The procedure and results are summarized in
 160 Appendix 1.

161



162

163 Figure 3 Schematic of the optical arrangement.

164

165 2.3.1. Laser Extinction Method

166 The Laser Extinction Method is based on the attenuation that a light beam undergoes when it
 167 traverses a soot cloud, which is quantified in terms of the Lambert-Beer's law as:

168
$$I = I_0 \exp(-K_{LEM}L) \quad (1)$$

169 where I and I_0 are the attenuated and original intensities, K_{LEM} is the dimensional extinction
170 coefficient of the cloud of particles and L is the path length, which corresponds to the size of the
171 cloud in the direction of the light beam. The extinction coefficient can be expressed as:

$$172 \quad K_{LEM} = \frac{k_{soot} f_v}{\lambda} \quad (2)$$

173 where f_v is the soot volume fraction, λ is the laser wavelength and k_{soot} is the dimensionless
174 extinction coefficient. The optical arrangement set for the Laser Extinction Method is shown in
175 Figure 3. A continuous Argon laser was set to cross the combustion chamber through two aligned
176 optical accesses. The laser was tuned at 514.5 nm with 400 mW and oriented with a 1 degree
177 angle of incidence in relation to the entrance quartz window due to space and optics limitations.
178 Besides, it was observed that this orientation made it possible to remove any influence of the
179 etalon effect on the measurements [30]. In order to minimize the divergence of the laser, a 500
180 mm focal length lens was set just at the output of the fiber optics that were used to guide the
181 beam from the laser output towards the test rig. The minimum beam waist (300 μm diameter)
182 was located in the region of the flame and the laser beam was aligned in a way that it was
183 crossing the flame axis. Once the laser left the combustion chamber, it was reflected by a beam
184 splitter (60% Transmission – 40% Reflection) towards the collection optics. .

185 Musculus et al. [30] present an extended analysis of several uncertainty sources that must be
186 considered when LEM is applied. Two major issues were identified: beam steering and the
187 contamination of measurement by light coming from the flame. The first one is a consequence
188 of the refractive index gradients inside the combustion chamber due to fuel evaporation and
189 combustion. In order to minimize this effect, a 50 mm diameter lens was placed just after the
190 beam splitter, to collect deviated rays up to a maximum divergence angle of 150 mrad. If the
191 maximum divergence angle collected is too large, the light emitted by the flame can be also
192 registered leading to an underestimation of the light extinction. In this sense, a diaphragm was
193 located at the focal plane of the lens to limit the maximum divergence angle to 100 mrad [30].
194 Finally a bandpass filter was placed between the diaphragm and the detector (centered at 514
195 nm with 10 nm FWHM) to reject the major part of the flame radiation. The detector is a fast
196 response photodiode, connected directly to an integrating sphere.

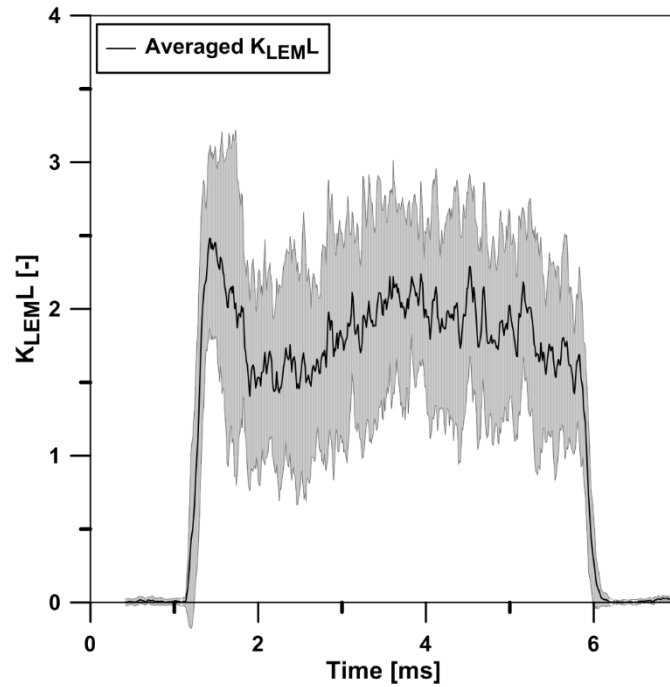


Figure 4 Average $K_{LEM L}$ and standard deviation for $P_c = 5.3$ MPa, $T_c = 900$ K and $P_{inj} = 100$ MPa, at 50 mm from nozzle tip.

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200 Light extinction was measured at several positions along the spray axis: 33 mm, 42 mm, 51 mm
 201 and 60 mm for two nominal conditions ($P_c = 5.3$ MPa, $P_{inj} = 100$ MPa and the two in-cylinder
 202 temperatures), and only at 33 mm and 51 mm for the rest of conditions. In order to calculate
 203 the instantaneous transmissivity of the flame, the attenuated intensity from each combustion
 204 event is compared with the intensity registered in the previous motored cycle (equation -1-).
 205 This ensures that effects like window fouling or intensity variations from the laser do not affect
 206 measurements. In Figure 4, an example of $K_{LEM L}$ evolution is presented. The black line shows the
 207 $K_{LEM L}$ averaged between 15 repetitions. The grey area represents is limited by \pm one standard
 208 deviation, which is not negligible. A similar behaviour was previously reported by Payri et al.
 209 [31], where the authors analyse the scattering inherent to the test rig.

210 2.3.2. 2-Color Pyrometry

211 2-Color Pyrometry is based upon recording of spontaneous soot incandescence. The intensity of
 212 such radiation source (I_{soot}) is equal to the product of the radiation emitted by a black body at
 213 the same temperature (T) and the emissivity of the particles, which can be expressed in terms
 214 of soot concentration, working wavelength (λ) and a dispersion exponent (α) [22]. Therefore,
 215 I_{soot} can be expressed as the following equation:

$$216 I_{soot}(\lambda, T, KL) = \left[1 - \exp\left(-\frac{K_2 c L}{\lambda^\alpha}\right)\right] \frac{1}{\lambda^5} \frac{c_1}{\left[\exp\left(\frac{c_2}{\lambda T}\right) - 1\right]} \quad (3)$$

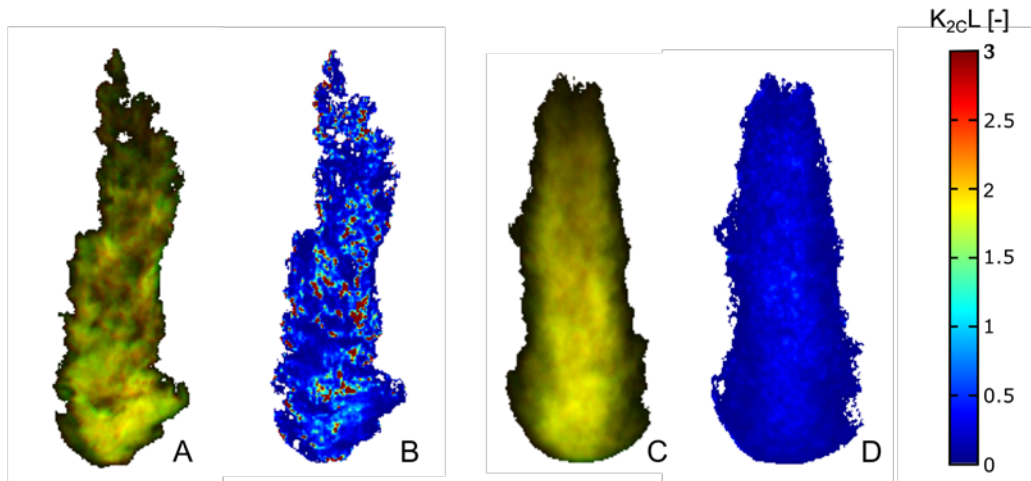
217 where $c_1 = 1.1910439 \times 10^{-16} \text{ Wm}^2\text{sr}^{-1}$ and $c_2 = 1.4388 \times 10^{-2}\text{mK}$. Zhao et al. [19] reported that α
218 is less dependent on the wavelength in the visible range than in the infrared. According to that,
219 550 and 650 nm have been chosen for this work, so that $\alpha = 1.39$ for most of the fuels [34]. The
220 dependence of the emissivity on the soot amount within the optical path is usually expressed in
221 terms of K_{2cL} . This variable accounts for the total contribution of the soot along the optical path,
222 no matter either the soot distribution or geometrical size.

223 Two CMOS sensors were employed to measure soot radiation. The signal recorded signal S_λ can
224 be expressed after several simplifications can be applied [22] according to equation (4):

$$225 \quad S_\lambda = C_\lambda I_{soot}(\lambda, T, KL) \quad (4)$$

226 where C_λ is a constant that quantifies the effects of the area A of the sooting flame within the
227 field of view of the detector, the solid angle Ω and the wavelength λ . The two first parameters
228 being constant, C_λ is calculated by means of a radiance calibration procedure as described by
229 Payri et al. [22].

230 In the setup shown in Figure 3, light emitted by the sooting flame crossed a first beam splitter
231 (60% transmission – 40% reflection), which was placed to reflect the LEM laser. Then, a second
232 beam splitter is used to transmit and reflect 50% of the soot radiation to each of the two high-
233 speed CMOS cameras employed (Phantom V12 for 650 nm, and Photron SA5 for 550 nm). Both
234 cameras were equipped with a 100 mm focal length and $f/2$ lens and an interference filter,
235 centred at 650 nm and 550 nm respectively with 10nm FWHM. Images were recorded at 15000
236 fps, with 5 to 8 us exposure time for 650 nm and 8 to 12 us for the 550 nm, depending on test
237 conditions. To match both images on a pixel by pixel basis, a spatial transformation matrix is
238 calculated, considering translation, rotation and scaling. For both images, background
239 segmentation is also applied. A threshold value is obtained, considering a percentage of the total
240 dynamic range of each image. The value of this percentage was set to 5% for all the tests, which
241 has shown a good accuracy on the flame boundary detection for all the tests.



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Figure 5 Composition of instantaneous (A) and average (C) soot natural luminosity at 550 and 650 nm and the corresponding K_{2cL} (B and D) distributions. Data were taken for n-Decane, at $P_c = 5.3$ MPa, $T_c = 900$ K and $P_{inj} = 100$ MPa.

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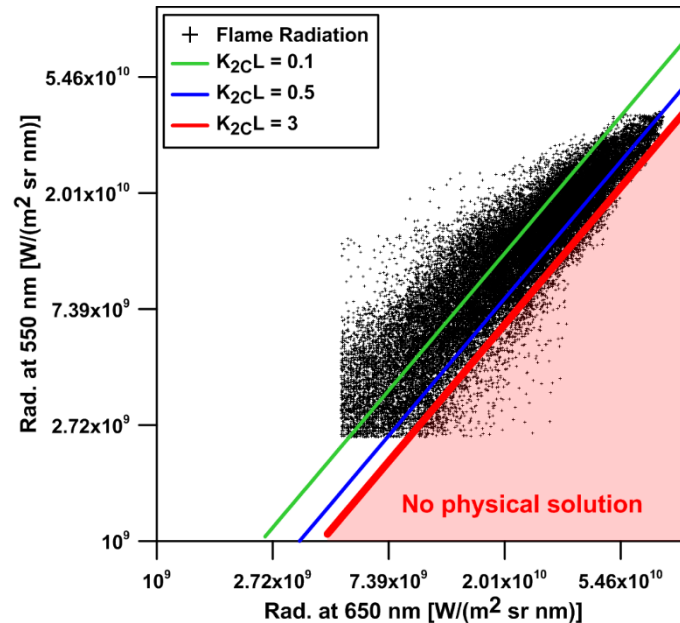
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Once both images are coupled, equation (3) is applied for each wavelength and K_{2cL} and temperature can be obtained. In Figure 5, an example of the application is shown. A colour composition of the instantaneous soot natural luminosity for 550 nm and 650 nm (A) is presented, together with the calculated map of K_{2cL} (B). It is possible to see that the K_{2cL} distribution is not homogeneous and even some spots of constant $K_{2cL} = 3$ are observed. These spots are artificially introduced, and are formed by pixels where the combination of radiation at the two wavelengths leads to a non-physical solution. In Figure 6, radiance values at 550 and 650 nm for each pixel of the flame shown in Figure 5 are presented. Moreover, three curves are plotted which represent the different combinations of radiation that lead to $K_{2cL} = 0.1$ (green), $K_{2cL} = 0.5$ (blue) and $K_{2cL} = 3$ (red). When K_{2cL} increases, its emissivity tends asymptotically to 1 (black body). For $K_{2cL} = 3$, the corresponding emissivity at 550 nm is 0.999. Therefore, the red curve can be interpreted as a frontier of the 2C methodology. Different uncertainty sources [21, 22] can lead to a combination of intensities of radiation located in the red area of Figure 6, which leads to a non-physical solution. When this happens, it is not possible to obtain a value of K_{2cL} for such pixels and maximum $K_{2cL} = 3$ is assigned. A similar heterogeneous distribution was previously reported by other authors [21, 26, 27]. Svensson et al. [21] suggest that the heterogeneity is real and not caused by uncertainty sources. However, Payri et al. [22] conclude that their influence is not negligible, leading to variations of K_{2cL} up to 20%. To minimize the effect of such non-physical pixels, the 2C method is applied to the ensemble-averaged imaged at each time position (Figure 5 - C), similar to Yan et al. [17]. Corresponding K_{2cL} results are presented in Figure 5 - D. In that way, the influence of measurement uncertainties such as read

267 noise are minimized. The KL distribution shown in Figure 5 – D also shows that the number of
268 pixels with non-physical solutions are reduced.



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Figure 6 Intensity of radiation for each pixel of a flame, at 550 and 650 nm. Data was taken for n-Decane, at $P_c = 5.3$ MPa, $T_c = 900$ K and $P_{inj} = 100$ MPa.

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274 2.3.3. Laser-Induced Incandescence

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Laser-Induced Incandescence is based on the thermal radiation emitted by a soot cloud, when it is irradiated with an intense laser pulse that increases its temperature, with a corresponding increase in local radiation. In the present contribution, a laser sheet was created to heat the soot particles at the symmetry plane of the flame. A Nd:YAG laser pulse at 1064 nm was used, with 600 mJ/pulse and a Gaussian intensity profile. The main wavelength of this laser was chosen to avoid the PAH fluorescence, as it has been previously reported by Bobba et al. [4]. Three cylindrical lenses were used to obtain a 45 x 0.35 mm² collimated laser sheet, with 450 mJ/pulse at the entrance of the combustion chamber. The equivalent energy fluence is 2.87 J/cm², which is large enough to get a signal independent of the laser pulse energy as it has been previously reported by other authors [4, 13, 16]. However, care must be taken during analysis as this high energy fluence could evaporate the smallest soot particles. The laser sheet was located to obtain LII signal from 22 to 67 mm from the injector nozzle, covering a similar range as the LEM measurement. The signal was registered by a 16-bit intensified CCD camera (LaVision Dynamight), equipped with a 100 mm focal length and f/2 UV lens. A low pass filter with the

289 cutting wavelength at 400 nm was placed in front of the detector, to improve the separation
290 between LII and natural luminosity from the flame. Nevertheless, for each test, five background
291 radiation images were recorded, ensemble-averaged and subtracted from the LII signal. Such
292 background levels were around 10% of the LII signal. It must be noted that this procedure is not
293 an instantaneous correction. Therefore, it can be introducing local errors when single repetitions
294 are considered. A 50 ns gate width was chosen, to minimize the influence of ambient conditions
295 and possible electronic jitter between the laser and the camera [13].

296

297 **2.4. Evaluation strategy for soot quantification techniques**

298 One of the main goals of the present contribution is a comparison among all three diagnostic
299 techniques. For that purpose, KL values (optical thickness) have been chosen as metrics of soot
300 measurements [6, 30, 31, 35]. In this sense, a discussion regarding soot optical properties is
301 avoided. According to the literature survey, LEM measurements have been chosen as reference,
302 so performance and limitations of 2C-Pyrometry and LII can be analysed relative to this
303 technique.

304 Under the assumption of thermal equilibrium, the emissivity of the soot cloud equals its
305 absorptivity (Kirchhoff's law). Moreover, if the interaction between light and soot particles is in
306 the Rayleigh regime, absorption would be dominant and scattering could be disregarded.
307 Therefore, the emissivity of the 2-Color Pyrometry and LEM absorption can be compared as
308 follows:

$$309 \quad 1 - \exp(-K_{LEM}L) = 1 - \exp\left(\frac{-K_{2C}L}{\lambda^\alpha}\right) \quad (5)$$

310 where K_{LEM} is the optical thickness obtained by means of LEM. To compare both techniques,
311 the same physical magnitude has to be used. At this point, nomenclature in the literature is
312 inconsistent, as both extinction and 2C derived results are defined as KL. Therefore, $K_{2C}^* =$
313 K_{2C}/λ^α has been defined, which should enable a direct comparison between 2C and LEM, i.e.
314 $K_{2C}^* = K_{LEM}$. Hence, considering that the wavelength used for laser extinction was 514.5 nm
315 and $\alpha = 1.39$, the following relationship can be derived in this case:

$$316 \quad K_{LEM}L = K_{2C}^*L = 2.519 \cdot K_{2C}L \quad (6)$$

317 which allows comparing the optical thickness of the flame obtained by means of LEM with 2C
318 measurements. Note that the right-hand side of equation (5) is a semi-empirical derivation
319 where λ is to be expressed in μm .

320 On the other hand, the starting point of LII technique is the assumption that the recorded signal
321 is proportional to the soot volume fraction [19], equation (7):

$$322 \quad f_v = C \cdot I_{LII} \quad (7)$$

323 where f_v is the soot volume fraction, C is a constant and I_{LII} is the registered LII signal intensity at
324 each pixel. The calculation of C can be addressed in two different ways. The first one is based on
325 numerical approaches to characterize all the physical phenomena involved in the process.
326 Different theoretical models can be found in the literature [10]. However, all of them show high
327 complexity, especially under engine conditions. The second procedure is based on an empirical
328 calibration by means of an additional experimental technique [11-16]. Despite certain
329 limitations, results suggest that at least some semi-quantitative results on the soot distribution
330 can be obtained. Following this approach, if equation (2) and (7) are combined, the following
331 expression can be obtained:

$$332 \quad K_{LEM} = C \frac{k_{soot}}{\lambda} \cdot I_{LII} = \frac{1}{C^*} \cdot I_{LII} \quad (8)$$

333 As the comparison between LII and LEM has to be done in integrated values along the optical
334 path, an integrated LII signal S_{LII} is defined as:

$$335 \quad S_{LII} = \int_0^L I_{LII} dx \quad (9)$$

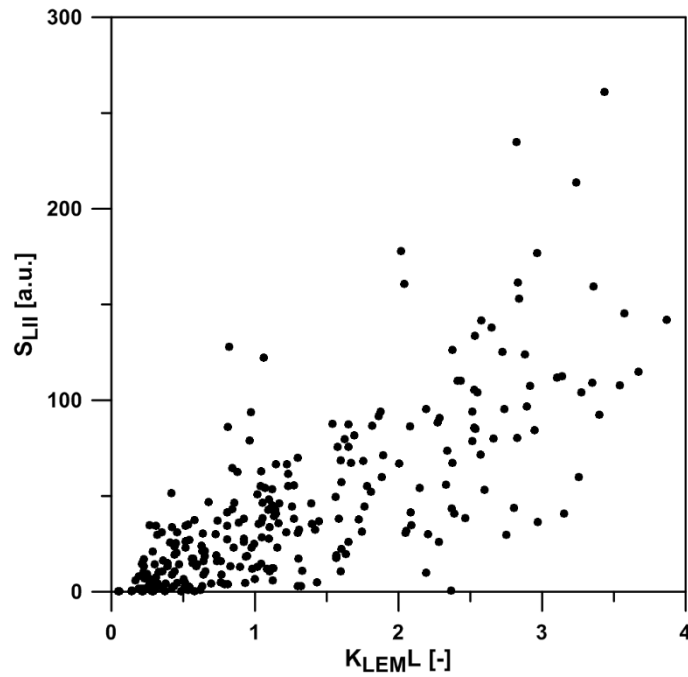
336 and by integration of equation (8) the following expression is obtained:

$$337 \quad S_{LII} = C^* K_{LEM} L \quad (10)$$

338 As previously discussed, the calibration constant C^* has been calculated by comparing $K_{LEM}L$
339 measurements with the corresponding I_{LII} integrated along the optical path (the width of the
340 flame). Once the calibration constant is obtained, a LII-derived optical thickness for $\lambda = 514.5$
341 nm has been calculated ($K_{LII}L$) to enable a comparison with LEM and 2C results according to
342 equation (11).

$$343 \quad K_{LII}L = \frac{1}{C^*} S_{LII} \quad (11)$$

344 In Figure 7, the comparison between S_{LII} and $K_{LEM}L$ for n-Decane is shown. 360 different
345 measurement points are plotted which include all the different test conditions and the LEM
346 measurement positions. $K_{LEM}L$ values correspond to the average of the last 50 μs just before the
347 LII laser is fired, to minimize scattering caused by the measuring technique (Figure 4).



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Figure 7 Correlation between K_{LEML} and S_{LII} for n-Decane. Single shot values.

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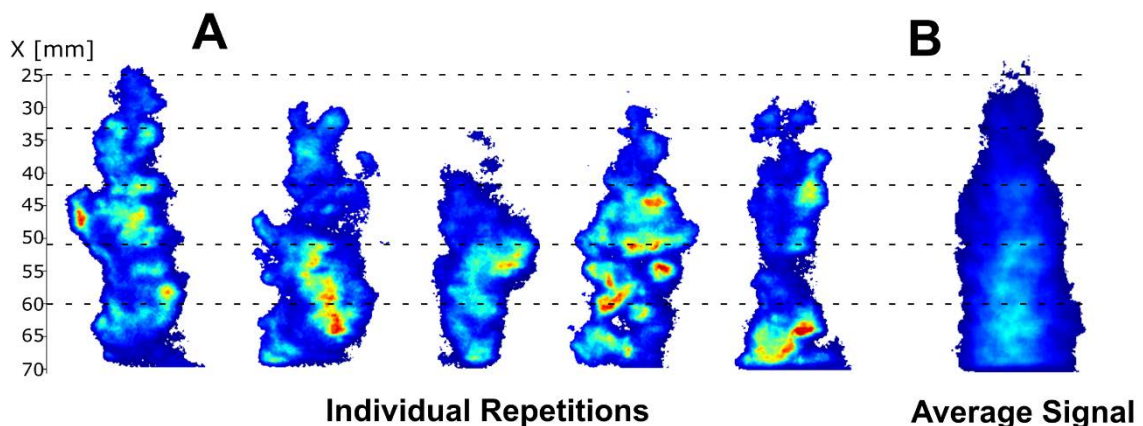
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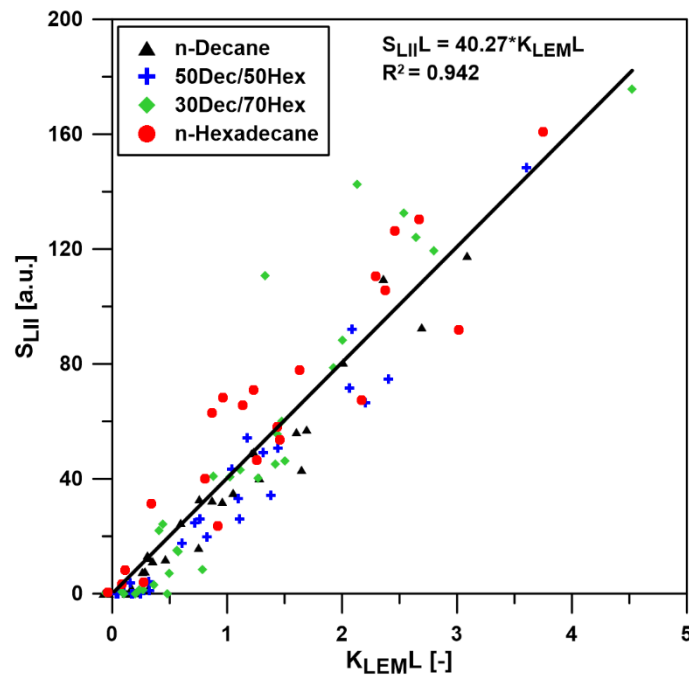
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Figure 8 Comparison between individual repetitions and the averaged signal for n-Decane, at $P_c = 5.3$ MPa, $T_c = 900$ K and $P_{inj} = 100$ MPa. The dashed lines represent the positions where laser extinction was measured.

361 In Figure 8 (A), single-shot LII images for different n-Decane injections are shown. Scattering is
 362 clearly present among different repetitions and, what is more, the flame is not perfectly
 363 axisymmetric. The authors consider that this behaviour is inherent to the operating conditions
 364 of the test rig, caused by the interaction between the spray and the air flow inside the
 365 combustion chamber, which was previously analysed by Payri et al. [31]. In that work a strong
 366 scattering observed on LoL and K_{LEM} measurements was also reported. However, if the
 367 ensemble-averaged LII signal is observed (Figure 8 -B-), the assumption of axisymmetry still
 368 seems to be reasonable. Following this approach, Figure 9 shows the same relationship as Figure
 369 7 based on ensemble-averaged values (sample size 75 repetitions for nominal points, i.e. $P_c =$
 370 5.3 MPa and $P_{inj} = 100 \text{ MPa}$, and 30 for other conditions). Data from all fuels have been included.
 371 Based on a linear regression ($R^2 = 0.942$) the calibration constant C^* for the whole data set can
 372 be obtained, which is independent of fuel and in-cylinder conditions.



373

374 Figure 9 Comparison between K_{LEM} and S_{LII} for the four fuels. Ensemble-averaged values.

375 Summing up, individual cycle realization show hardly any correlation between the LEM and LII
 376 signals, but when using ensemble-averaged ones the situation improves. Based upon previous
 377 experiences with this experimental setup [31], where a strong cycle-to-cycle is present, coupled
 378 to the 1° inclination angle between the LEM point laser and the LII laser sheet could be a strong
 379 reason why a single realization comparison may not be fully correlated. When using ensemble-
 380 averaged values, the assumption of axisymmetry is recovered, and the correlation between
 381 both signals improves. However, some scattering is still present, which hints at the presence of

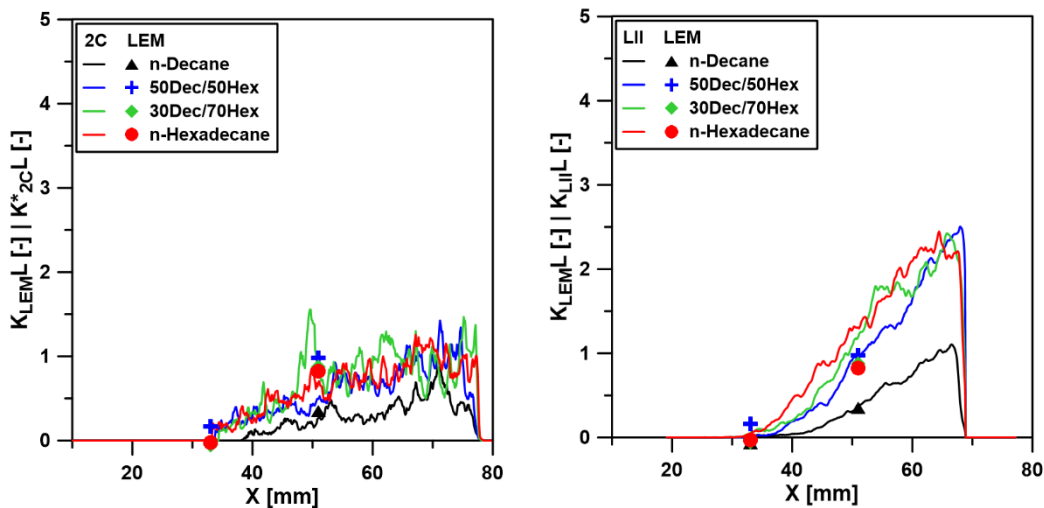
382 absorption phenomena. Therefore LII results should be used in a semi-quantitative way. More
 383 discussion on such effects will be shown in Section 4.

384

385 3. FUEL EFFECTS ON SOOT DISTRIBUTION

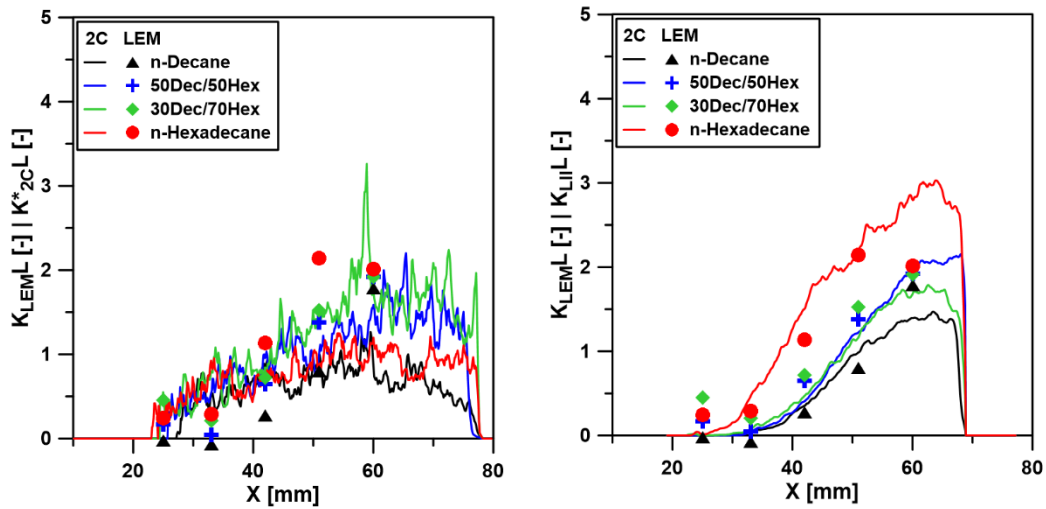
386 LEM, 2C and LII have been utilized to study the effect of fuel properties over soot formation.
 387 However, a direct comparison between the different techniques also makes it possible to
 388 determine reliability and limitations of each of them. As previously mentioned, LEM has been
 389 chosen as reference and is compared with 2C and then LII. From Figure 10 to Figure 12, KL
 390 measurements on the flame axis are presented for the four fuels at three different ambient
 391 conditions, which have been selected from the test matrix as representative of low ($P_c = 4.3$ MPa,
 392 $T_c = 800$ K), medium ($P_c = 5.3$ MPa, $T_c = 800$ K) and high ($P_c = 5.3$ MPa, $T_c = 900$ K) sooting conditions.
 393 K_{LEM} values correspond to the average of the last 50 μs just before the LII laser is fired. $K_{2C}^* L$
 394 values correspond to the 2C results obtained at 3 CAD after TDC, coinciding with the LII
 395 measurement. For each LEM position, 15 LII images were recorded, which means that the LII
 396 signal is averaged from 75 different images for the nominal points ($P_c = 5.3$ MPa and $P_{inj} = 100$
 397 MPa) and 30 for the rest of the tests matrix. K_{LII} is derived by means of equation (11).

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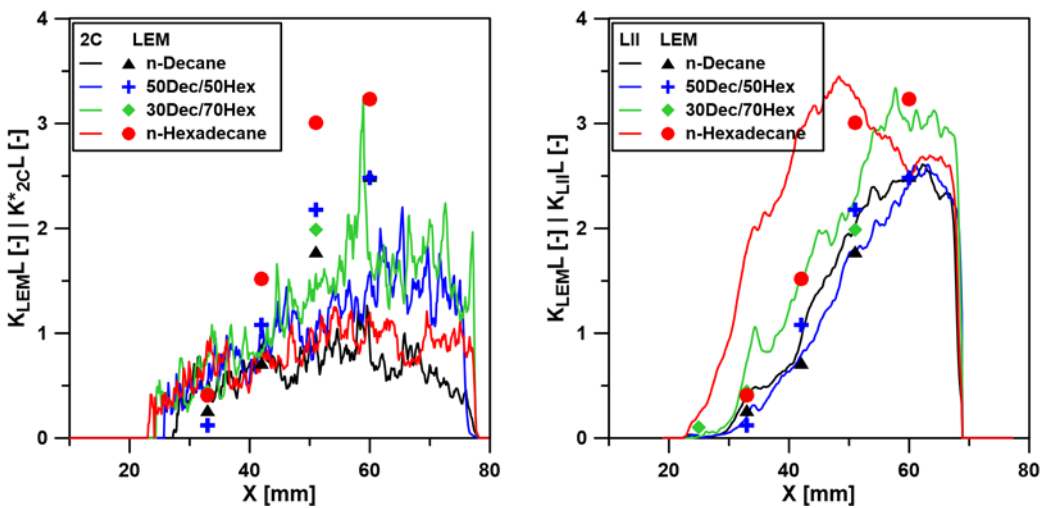
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400 Figure 10 K_{LEM} , $K_{2C}^* L$ (left) and K_{LII} (right) on the flame axis, for the four fuels at 800 K in-cylinder temperature,
 401 $P_c = 4.3$ MPa and $P_{inj} = 100$ MPa.



402

403 Figure 11 $K_{LEM,L}$, $K_{2C,L}^*$ (left) and $K_{LII,L}$ (right) on the flame axis, for the four fuels at 800 K in-cylinder temperature,
 404 $P_c = 5.3$ MPa and $P_{inj} = 100$ MPa.



405

406 Figure 12 $K_{LEM,L}$, $K_{2C,L}^*$ (left) and $K_{LII,L}$ (right) on the flame axis, for the four fuels at 900 K in-cylinder temperature,
 407 $P_c = 5.3$ MPa and $P_{inj} = 100$ MPa.

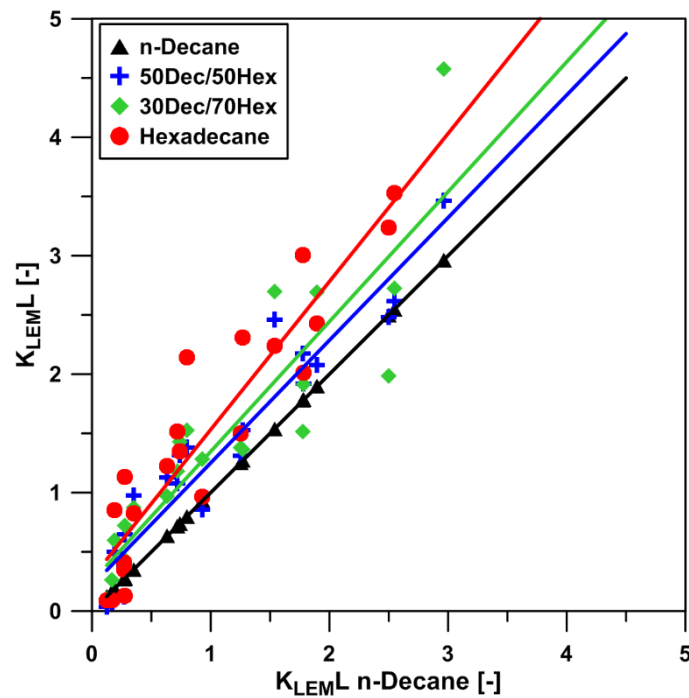
408 If LEM is used as a reference, soot is observed to effectively increase with ambient density. Such
 409 a result is consistent with literature studies [29, 31], and can be justified based on the reduction
 410 in lift-off length (LoL) that occurs with ambient density, which increases the equivalence ratio at
 411 the flame base, and hence induces higher soot formation. LoL values derived from OH*
 412 chemiluminescence images recorded simultaneously with the soot measurements (Appendix 1)
 413 confirm such a reduction for the present test matrix.

414 On the other hand, for a given operating condition LEM measurements evidence that the
 415 increase in n-Hexadecane content results in more soot being formed. Two main factors
 416 contribute to such a trend. On the one hand, simultaneous OH* measurements for this fuel
 417 sweep (Figure 17) indicate that LoL is reduced with the increase of n-Hexadecane, due to the
 418 increased reactivity of this fuel (higher CN). As the mixing field is quite similar for all investigated

419 fuels, this results in an increase of the equivalence ratio at the flame base, which enhances soot
420 formation. On the other hand, the Threshold Sooting Index (TSI) is also observed to increase
421 when shifting to larger alkanes [37], which means that the fuel is more prone to form soot.

422 A summary of the aforementioned trends with fuel composition is presented in Figure 13. K_{LEM}
423 for n-Decane is compared with the other three fuels for all investigated conditions. Although
424 some scattering is present, such a plot confirms the observed result in Figures 10 to 12.

425



426

427 Figure 13 Comparison between K_{LEM} of n-Decane and the other three fuels.

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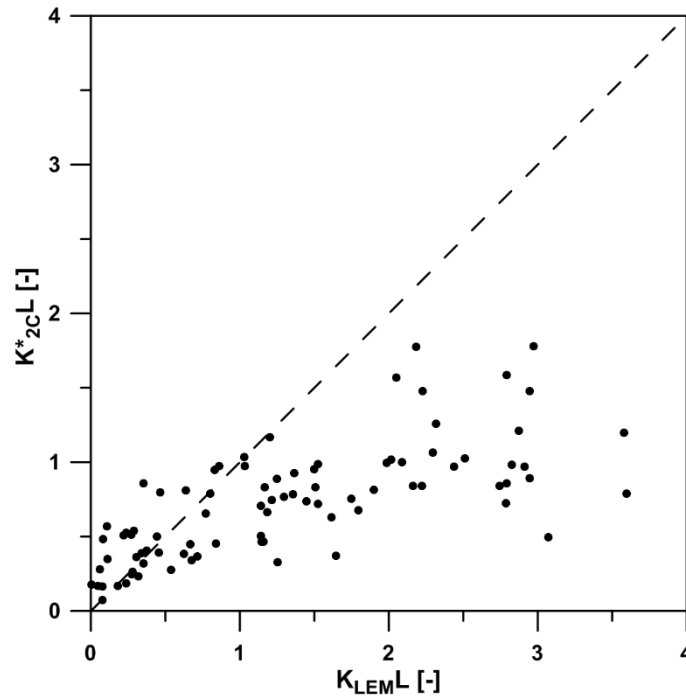
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430 4. DISCUSSION ON SOOT DIAGNOSTICS

431 The previous section has shown the spatially resolved results obtained for the investigated
432 conditions and fuels keeping LEM as the reference technique. This section will provide further
433 discussion on the other two optical techniques.

434 On the one hand, 2C is observed to be quite insensitive to operating conditions, especially when
435 shifting from medium to high density conditions (Figures 11 and 12). Even for a single operating
436 condition, the increase of $K_{2C}^* L$ along the spray axis is quite modest in comparison with K_{LEM}
437 evolution. In terms of fuel, only decane is consistently in the low range of measured values, while

438 the trends among the other fuels are difficult to discern. Both statements confirm that, if any
 439 sensitivity in 2C is to be obtained, it mainly occurs in the low soot range. Figure 14 summarizes
 440 these findings on a one-to-one comparison between techniques. In general, it can be stated that
 441 values provided by 2C are lower than the ones obtained by LEM. Furthermore, these differences
 442 are shown to increase when the optical thickness of the flame increases (larger $K_{LEM}L$). Svensson
 443 [38] also reported differences in experimental optical thickness from the two techniques, with
 444 maximum values between 4.49 and 2.66 for $K_{LEM}L$, and between 1.3 and 1.02 for K_{2C}^*L .

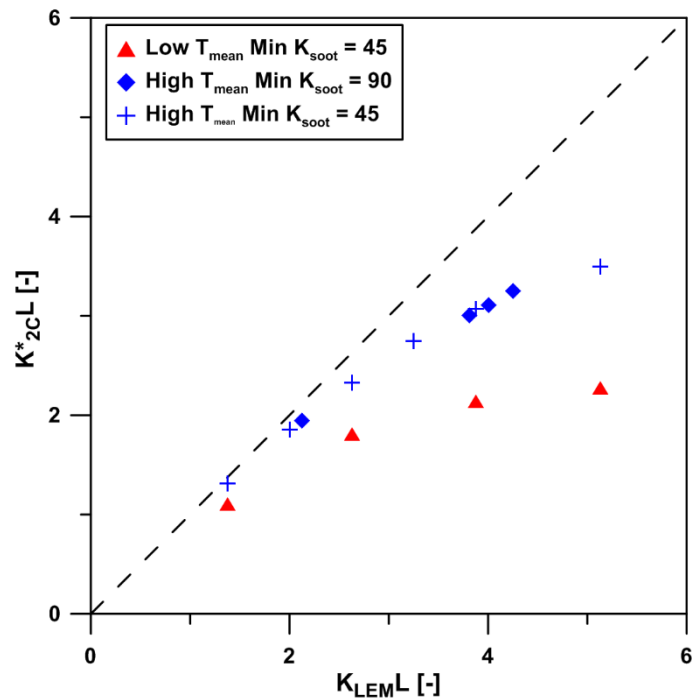


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Figure 14 Comparison between experimental $K_{LEM}L$ and K_{2C}^*L .

447 A theoretical approach has been followed to better understand the behaviour of 2C technique.
 448 This analysis, which is described in Appendix 2, is based on dividing the flame profile in finite
 449 elements with defined k_{soot} and temperature. An accumulated radiated intensity is calculated
 450 for 650 and 550 nm and equation (3) is applied to obtain the corresponding $K_{2C}L$. At the same
 451 time, the optical thickness of the profile is calculated, which corresponds to $K_{LEM}L$. For different
 452 k_{soot} and temperature distributions (Figure 18), the corresponding K_{2C}^*L and $K_{LEM}L$ have been
 453 obtained. Three data sets are presented in Figure 15. For each of them, the temperature profile
 454 and the value of $Min K_{soot}$ were kept constant, while $Max K_{soot}$ was modified to simulate the
 455 variation in the total soot mass within the flame. Values of $Max K_{soot}$ have been varied between
 456 45 ($f_v = 13$ ppm) and 370 ($f_v = 50$ ppm).



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Figure 15 Theoretical comparison of $K_{LEM}L$ and K_{2C}^*L for different k_{soot} and temperature distributions.

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Theoretical calculations show a trend that is consistent with the experimental results presented in Figure 14. For the high T_{mean} case, no differences can be observed in K_{2C}^*L when soot distribution shape is modified at constant $K_{LEM}L$. This suggests that the total amount of soot is more important than its distribution along the flame width. However, when the total soot amount changes ($K_{LEM}L$ increases) at constant T_{mean} , K_{2C}^*L changes with a much lower sensitivity. This means that absorption effects within the optical path become relevant, and a fraction of the intensity emitted by the different soot layers does not reach the detectors. The difference between KL values from both techniques ranges from 20% for $K_{LEM}L \approx 1$ to almost 80% for $K_{LEM}L \approx 4$.

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On the other hand, if both temperature profiles are compared at constant soot distribution, an increase in K_{2C}^*L with T_{mean} is observed. This result agrees with recent measurements by Skeen et al. [39], where the ratio between KL as derived from LEM and spectral radiation measurements range from 1.5 at the highest temperature to 5 at the lowest temperature. Some explanation can be found by using the radiation propagation model. For that purpose, one has to keep in mind that the measured radiation is the result of the emitted radiation that propagates through the soot cloud and therefore becomes attenuated. The emission term depends on local temperature and soot amount, while the absorption one on the soot distribution. The measured radiation (and therefore temperature and KL factor) is therefore a

478 weighted value along the line-of-sight. Derived results coincide with the real ones only if a
479 uniform temperature distribution exists. If this is not the case, emitted radiation is biased
480 towards the hottest part of the soot cloud, because of the non-linear temperature dependence
481 of Planck's law. Considering the temperature variation in the previous plot, the high T_{mean} case
482 has a more uniform temperature distribution, which results in a better agreement between
483 K_{2C}^*L and $K_{\text{LEM}}L$. When going to the low T_{mean} case, temperature gradients increase within the
484 flame, and the influence of the outer (hotter) layers over the final measured radiation is more
485 important than in the high T_{mean} case. This leads to larger differences between the soot as
486 measured from the 2C and the real soot. Such effects decrease when soot concentration is low,
487 as attenuation effects decrease and the contribution of the inner layers can still be maintained.
488 Therefore, care must be taken when using results obtained from 2-Color Pyrometry under highly
489 sooting conditions.

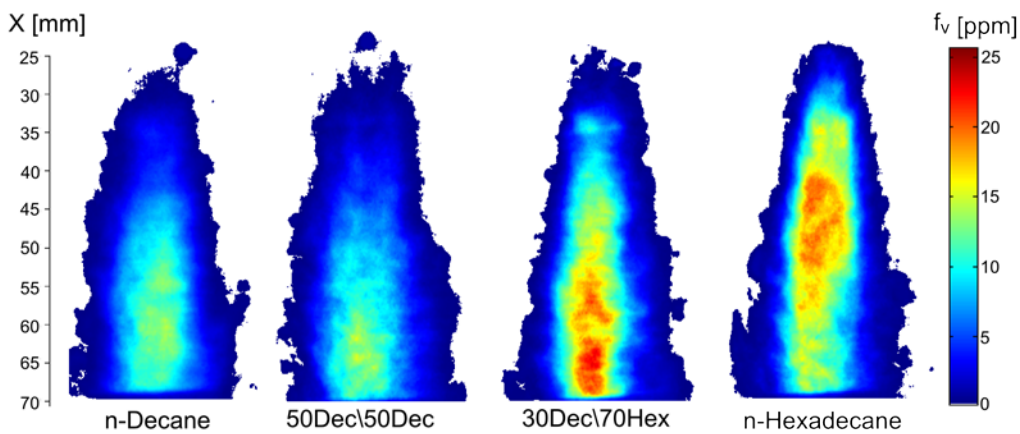
490 Similar arguments can be found in Musculus et al. [23] when comparing CFD calculations and
491 experimental measurements of integrated radiation from the whole combustion chamber.
492 These and other experimental references hint at the same conclusions, namely that 2C
493 thermometry cannot resolve the full soot amount within the flame.

494 On the other hand, results in the previous section has shown that LII has a sensitivity to operating
495 conditions and fuels comparable to LEM. However, some inconsistencies are present regarding
496 the one-to-one comparison between LEM and LII, especially for the highest sooting operating
497 condition, where a high discrepancy is found for hexadecane. It is known that LII can be strongly
498 affected by phenomena like signal trapping or laser light attenuation [10], which would lead to
499 a lack of LII signal at certain areas of the flame. This effect would be observable when $K_{\text{LEM}}L$ and
500 S_{LII} were compared, as a deviation from linearity. De Francqueville et al. [13] have used this
501 information to identify a set of experimental conditions where signal trapping is taking place,
502 namely the situation where low LII signal is detected but high $K_{\text{LEM}}L$ is measured. In the present
503 work, few cases show this behaviour (Figure 9). On the one hand, $K_{\text{LEM}}L$ was measured up to 60
504 mm from the nozzle orifice only for the reference points, while up to 50 mm for the rest of test
505 conditions. Moreover, LII signal is triggered at a timing where the flame is in a quasi-steady state.
506 Therefore, the high soot vortex is out of the field of view, and the high soot area is reached at
507 mid-way through the observation window. In this sense, considering the distributions observed
508 by the three techniques, the regions of maximum soot optical thickness were not measured with
509 LEM.

510 Still, some cases exhibit the soot attenuation effects. Figure 16 shows the LII-derived soot
511 volume fraction distribution from n-Decane (left) to n-Hexadecane (right) at $P_c = 5.3$ MPa, $T_c =$
512 900 K and $P_{inj} = 100$ MPa, corresponding to the evolution shown in Figure 12. The general aspect
513 of the 2D distribution is similar, with an increasing soot volume fraction with axial distance from
514 the nozzle. When comparing fuels, n-Decane and 50Dec\50Hex show similarly low soot values.
515 However, moving closer to pure n-Hexadecane results in higher soot. In particular, an apparent
516 decrease of soot when moving from 30Dec to pure n-Hexadecane in regions downstream of 50
517 mm can only be explained in terms of strong attenuation processes within the flame. Moreover,
518 in this region, soot distribution for n-Hexadecane becomes non-symmetrical, with higher values
519 on the left side of the image. With the optical set up described previously, the laser sheet was
520 entering from the left side of the flames shown in Figure 16. Thus, the previous effect could be
521 interpreted as being due to either beam attenuation or signal trapping by soot particles, as it
522 has been previously reported that both phenomena have a similar effect [11, 13].

523 The previous analysis invalidates the application of Equation (10) for the highest sooting
524 condition due to strong signal trapping, which breaks the proportionality between LII signal and
525 soot volume fraction. However, in the explored conditions, such effects are only present at some
526 particular conditions, and the validity of LII as a semi-quantitative technique can be assumed as
527 a fair argument.

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529

530 Figure 16 Soot volume fraction distribution for the four fuels at $P_c = 5.3$ MPa, $T_c = 900$ K and $P_{inj} = 100$ MPa.

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532

533 5. CONCLUSIONS

534 Four different fuels were characterized in terms of soot formation. For this purpose, the three
535 most extended optical techniques were used: Laser Extinction Method, 2-Color Pyrometry and
536 Laser-Induced Incandescence. The three optical accesses available allowed applying the three
537 techniques simultaneously, so that results were directly comparable. All the results have been
538 analysed in order to determine the reliability and usefulness of each technique. The main
539 conclusions are:

- 540 • In comparison with $K_{LEM}L$, K_{2C}^*L presents in general lower values and its sensitivity to
541 thermodynamic conditions and fuel properties is reduced. Moreover, it has been
542 observed that K_{2C}^*L seems to saturate when flame optical thickness increases. A further
543 theoretical analysis suggested that the measurements are strongly influenced by soot
544 and temperature distribution within the flame. When soot concentration is reduced
545 ($K_{LEM}L \leq 1$), 2C is still reliable.
- 546 • Laser-Induced Incandescence makes it possible to measure the soot distribution at any
547 plane within the flame, which is an advantage compared with line-of-sight diagnostics
548 such as LEM and 2C. A calibration procedure based on the combination of LII signal and
549 LEM measurements has been evaluated. The methodology has shown enough
550 sensitivity to characterize the influence of the different experimental parameters.
551 However, the beam attenuation and signal trapping effects have been observed to
552 strongly influence on the measurements for the highest soot conditions and locations.
553 Except for such cases, for most of the conditions LII can still be used as a semi-
554 quantitative measurement technique.

555 LEM and LII have shown to be accurate enough to characterize the differences in soot formation
556 for the four fuels considered in this study. It has been observed that the larger the molecule the
557 more soot is formed. This results from a concurrent reduction in lift-off length, which implies
558 lower oxygen entrainment at the flame base and therefore higher soot formation. A second
559 important effect, though, is the inherent sooting tendency of the fuel type (TSI index), which
560 increases with the molecule size for the investigated cases.

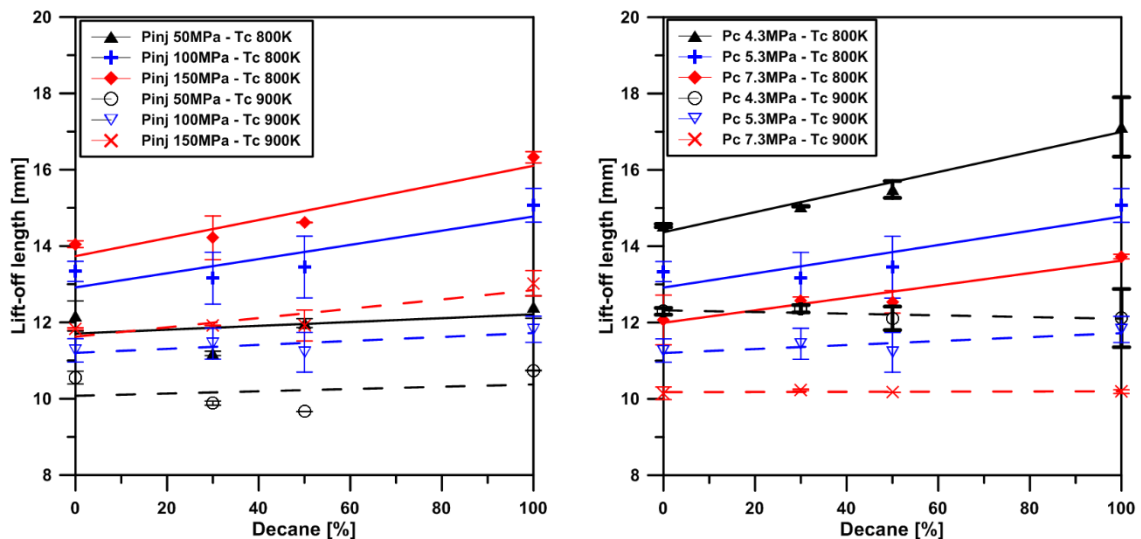
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563 APPENDIX 1: Lift-off Length measurements

564 Visualization of OH*-Chemiluminescence at the base of the flame makes it possible to quantify
565 the lift-off length (LoL). A gated 16-bit intensified CCD camera (Andor iStar) was utilized,
566 equipped with a UV f/4 100 mm focal length lens. An interference filter centred at 310 nm (10
567 FWHM) was placed in front of the camera to remove the major part of the radiation of the flame
568 while keeping OH*-Chemiluminescence. The camera was triggered at 6.6 after SoE while the
569 intensifier was gated during 2.4 CAD and the gain was set to use the complete dynamic range of
570 the camera without saturating it. Background segmentation was applied, based on a threshold
571 value calculated as a percentage of the dynamic range of each image. This percentage was set
572 to 10%, as it offered a good compromise for all the different test conditions. Then lift-off length
573 was defined as the average distance between the nozzle and the ten nearest pixels of the flame.

574 In Figure A-1 LoL vs. fuel composition is presented, for different injection pressures (left) and
575 in-cylinder pressures (right) f both in-cylinder temperatures. It can be observed that the LoL
576 increases with the n-Decane fraction, which is consistent with the change in fuel reactivity (lower
577 Centane number). LoL increases for all the fuels with injection pressure, while it decreases with
578 in-cylinder pressure. However, differences are seen to decrease when in-cylinder pressure and
579 temperature are increased. A similar effect is observed when the injection pressure is
580 decreased.



581
582 Figure 17 Lift-off length vs. n-Decane content, for different injection pressures (left) and different in-cylinder
583 pressures (right), at 800 and 900 K in-cylinder temperature.

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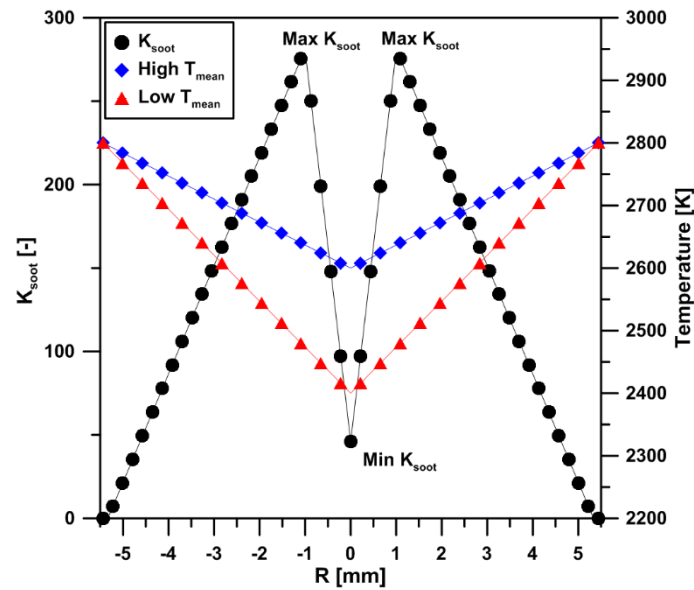
586 **APPENDIX 2 – Radiation propagation model**

587 Simple radiation propagation concepts have been used to improve the understanding of the
588 differences between K_{2cL} and $K_{LEM}L$, following a similar methodology as Payri et al. [22]. These
589 authors described the behaviour of the 2-Colour Pyrometry technique under different in-
590 cylinder conditions and compared experimental measurements with theoretical predictions,
591 based on the equations shown in the previous sections.

592 A flame profile, perpendicular to its axis, can be discretized as an axisymmetric distribution of
593 layers, with constant k_{soot} and temperature. Each of these layers both emits ($I_{soot,i}$) and absorbs (α_i)
594 radiation from the previous layers. Thus, the intensity measured by one detector on one side of
595 the flame is the result of an accumulation of emission and absorption processes along the optical
596 path, through all the layers inside the flame. $I_{soot,i}$ is calculated by equation (3) while α_i is
597 obtained from equation (5). Finally, it is possible to calculate the corresponding optical thickness
598 of the profile ($K_{LEM}L$) as the accumulation of the $K_{LEM}L_i$ from the different “i” layers inside the
599 flame as it is defined by equation (12).

$$600 \quad K_{LEM}L = \sum_{i=1}^{2R} 2.519k_{soot,i}L_i \quad (12)$$

601 Where R represents the maximum radius of the profile and L is the width of each layer. Several
602 flame temperature and k_{soot} radial distributions have been evaluated, based on previously
603 published experimental results [22], which are shown in Figure 18. It must be noted that this
604 approach assumes instantaneous oxidation of soot at the diffusion flame front. Therefore, radial
605 distribution of both Temperature and k_{soot} are only considered until that particular location. The
606 shape of k_{soot} profile was kept constant for all the calculations while the Min k_{soot} and Max k_{soot}
607 were modified to vary the total amount of soot. The temperature profile shape was kept also
608 constant, but the minimum value was modified in order to evaluate its influence on the final
609 measured K_{2cL} . Peak temperature was fixed at 2800 K, close to the values obtained at the edge
610 of the flame for $T_c = 900$ K cases.



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Figure 18 Theoretical radial profiles for k_{soot} and flame temperature distribution

613

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615

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