Messtechnikweiterentwicklung im Bereich der planaren Tropfengrößenbestimmung in Sprays basierend auf dem LIF/Mie-Verhältnis

Advances in planar droplet sizing in sprays based on laser-induced fluorescence and Mie-scattering

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Abstract

The present paper gives an overview on advances in planar droplet sizing (PDS) based on the ratio of laser-induced fluorescence (LIF) and Mie scattering for determination of the Sauter mean diameter (SMD). Recent measurements of the group are presented and a literature review is provided. Two different fluorescent dyes are studied in various polar and non-polar liquids and fuel mixtures. The individual droplets were studied at microscopic scale using a long-distance microscope and two cameras (for LIF and Mie-scattering, respectively). This setup is applied for calibration of the signals depending on droplet size, temperature and composition in a droplet chain. Finally, the calibration curves deduced are applied to extract the droplet SMD from instantaneous images of transient DISI (direct-injection spark-ignition) sprays.

Introduction

Technical sprays need to be characterized regarding droplet size, temperature and composition (Ashgriz 2011). This information is also required for development and validation of spray models and simulations. For this purpose, several non-invasive techniques were developed and optimized over the last decades. Laser-induced fluorescence (LIF) is an established technique for determination of droplet temperature and composition (Lemoine und Castanet 2013). Planar droplet sizing (PDS) for determination of the SMD (Sauter Mean Diameter) is based on the ratio of laser-induced fluorescence (LIF) and Mie scattering. This technique relies on the assumption that LIF and Mie scattering signals from spherical droplets depend on their volume and surface area, respectively (Le Gal et al. 1999, Domann und Hardalupas 2001, Domann et al. 2002, Domann und Hardalupas 2003, Frackowiak und Tropea 2010, Frackowiak und Tropea 2010, Charalampous und Hardalupas 2011, Koegl et al. 2018):

$$S_{LIF} = \alpha_{LIF} \cdot d^3_{droplet}$$

$$S_{Mie} = \alpha_{Mie} \cdot d^2_{droplet} .$$
(1)
(2)

The constants α_{LIF} and α_{Mie} contain experimental parameters such as the signal collection solid angle and the laser fluence. The absorbing mixture of the dye in the solvent leads to extinction effects, which affects the exponent in the LIF signal. A volumetric dependency is only ensured for low dye concentration (i.e. low absorbance). A high dye concentration (or high absorbance) leads to a decrease of the LIF exponent to 2 (Le Gal et al. 1999). The laser fluence has also an impact on the exponents (Frackowiak und Tropea 2010). The d²-dependency of the Mie signal is only valid for illumination with perpendicularly polarized light. Horizontal polarized light leads to a d¹-dependence (Massoli et al. 1989, Massoli et al. 1989). For Mie-scattering, it was found that the d² behavior is affected by the refractive index, the scattering angle, and dye concentration. The d² relation was best represented for a low dye concentration.

Besides effects of the dye concentration, also the temperature and laser fluence effects on LIF emissions must be characterized. The droplet temperature may change during evaporation. Besides a heating of the droplets in the hot ambience also a cooling may occur induced by the evaporation enthalpy of the fuel. Furthermore, also the dye concentration may vary drastically during droplet evaporation.

The appearance of morphologic dependent resonances (MDRs, or "lasing") have also an impact on the LIF-signal. MDRs are ring-shaped structures within the LIF signal. They are generated by the droplet itself, which acts as a laser cavity. The effects on MDRs in LIF/Mie measurements are not well characterized and could introduce large measurement uncertainties. MDRs occur at certain dye concentrations and high laser fluence, and depend on the solvent (Kwok und Chang 1993).

All these dependencies need to be studied for the respective dye and solvent and require a calibration. Several studies based on the LIF/Mie ratio have been conducted over the years mainly in water sprays, some in ethanol or fuel sprays. Most dyes used in the literature do not show solubility in automotive or aerospace fuels such as gasoline and Jet A-1. A promising dye for these fuels is nile red, which was applied for droplet sizing by the authors before (Koegl et al. 2019, Koegl et al. 2020). Various effects on the measurement accuracy of the LIF/Mie technique may occur. Not all are known in detail, but some of them are discussed below. For example, distinct effects of the solvents on the absorption and emission characteristics and signal intensity are possible, which could affect SMD measurements. The solvent (mainly pH-value and polarity) and the absorption and emission properties of the dye determine the LIF signal as well as MDR at the respective conditions.

In the present paper, an overview is provided on major parameters determining the LIF/Mie ratio and thus the accuracy of the technique, which is conducted for two main dyes suitable either for polar (such as ethanol or water) and non-polar fuels (such as alkanes).

Experimental Setup

In the following section, the setups for the calibration of the LIF/Mie ratio and the spray measurements are described. Calibration of the droplet signals was conducted using a droplet generator while the spray measurements were done for a DISI (direct-injection spark-ignition) injector in an injection chamber.

Droplet generator and microscopic LIF/Mie setup

A monodisperse droplet generator (MSP Corp, Type 1530) was utilized which is able to produce droplets ranging between 15 and 150 μ m. The flow focusing air and the fuel are kept at 298 K to enable constant conditions during the measurements at an ambient pressure of

0.1 MPa. The temperature of the droplet generator can be varied by using an in-house built heating jacket. The temperatures of the whole setup are monitored by integrated thermocouples (type K). The downstream distance between the droplet generator orifice and droplet measurement plane is between 3 and 7 mm, which depends on the droplet size.

The microscopic setup for calibration consists of a Nd:YAG-laser emitting a laser pulse at 532 nm. The LIF and Mie signal intensities are recorded with a single objective system, which is equipped with two identical scientific CMOS cameras (Imager, LaVision GmbH, Germany). Each recorded image corresponds to 2560 x 2160 pixels. The microscopic objective system is equipped with a long distance microscope (Infinity, model K2 DistaMax) with the region of interest equal to 0.71 mm x 0.60 mm and with a pixel resolution of 3.63 pixel/ μ m). The incoming signal is separated by using a cube beam splitter (70% transmission for the LIF signal and 30% reflection for the Mie signal). For generating the LIF signal, a dye is required that is added to the fuel as specified below.

The LIF emission is detected by using a 532 nm (17 nm FWHM) notch filter. The Mie scattering signal is detected by a 532 nm (1 nm FWHM) laser line pass filter.

Neutral density (ND) filters of different optical densities were applied to allow for the best possible signal to noise ratios at constant laser illumination.

Injection chamber and SLIPI LIF/Mie setup

A constant volume combustion (CVC) chamber is applied for investigation of DISI-sprays under engine-like conditions. It is operated with dry air at 0.2 MPa pressure and 298 K temperature. The ambient temperature in the CVC and the fuel temperature are set to 298 K. The injection duration is 1800 µs. The injection repetition rate was 0.5 Hz. The injection pressure is set to 16 MPa. A 5-hole DISI-injector (BOSCH) is utilized. It is characterized by one central jet that is separated from the other four jets allowing unrestricted optical access. Two laser pulses of 532 nm wavelength from an Nd:YAG laser cluster with top hat beam profiles were applied for 2p-SLIPI (2-Pulse Structured Laser Illumination Planar Imaging) in the spray chamber (details see Storch et al. (Storch et al. 2016)). The modulation is generated by a square Ronchi grating. This produces lines with 0.35 mm height with an equidistant spacing of 0.7 mm in the illuminated spray. The spatial phase shift is generated by a calcite crystal and two crosspolarized laser beams. The horizontally polarized laser pulse passes through the crystal. It maintains the same path (ordinary ray). The vertically polarized laser pulse is displaced (extraordinary ray). The spatial shift is determined by the thickness of the calcite crystal. The same cameras and filters as mentioned above were applied for macroscopic signal detection. A 135 mm objective (Pentacon 2.8/135) was utilized for detection enabling a pixel resolution of 0.15 pixels/µm.

Fuels and dyes investigated

The liquids under investigation are fuels such as ethanol and gasoline. Since gasoline or kerosene are multi-component fuels, a surrogate fuel "toliso" (consisting of isooctane and toluene) is applied and in some studies only isooctane.

Two luminescence dyes were applied for fluorescence studies: First, eosin-Y (Sigma Aldrich) is added with 0.5 vol. % to the liquid fuel ethanol. For toliso and iso-octane, the dye nile-red was applied, although it is also dissolvable in ethanol. However, the molar absorption coefficient is much lower for nile red compared to eosin Y, so that the dye concentration must be elevated for comparable SNR.

In Figure 1, the absorption and emission bands of eosin-Y are provided. The absorption band in the visible range is between about 450 nm and 550 nm. An excitation of eosin-Y at 532 nm leads to a broadband signal between 540 nm and 670 nm.

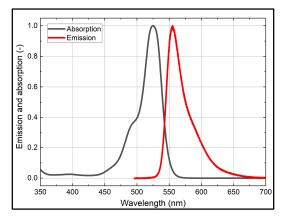


Figure 1: Absorption and emission spectra of eosin-Y in ethanol at 293 K.

The absorption and emission spectra of nile red dissolved in iso-octane ("E0") and E20 (i.e. a mixture of 20 vol.% ethanol and 80 vol.% isooctane) are provided in Figure 2. The absorption of nile red in isooctane occurs between 400 nm and 490 nm, the emission occurs between 500 and 750 nm. Both absorption and emission spectra of E0 show two distinct peaks. For E20, the absorption spectrum is broader and red-shifted, which is also true for the emission spectrum. Both spectra of E20 show just a single peak.

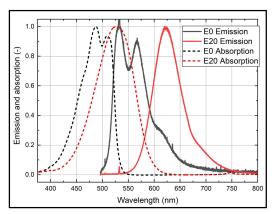


Figure 2: Absorption and emission spectra of nile red in E0 (pure isooctane) and E20 (20 vol.% ethanol and 80 vol.% isooctane) normalized to the respective maximum values. 293 K, 0.1 MPa. dye concentration: 3.5 mg/L.

Results

This section is divided into two parts. First, the potential of the PDS-technique based on the dye eosin is studied, which can be dissolved in polar liquids such as water and alcohols. Second, the behavior of nile red for planar droplet sizing is reviewed, which can be dissolved in non-polar liquids such as alkanes, although it can also be applied for polar liquids.

Calibration data and PDS in sprays based on eosin-Y for polar solvents

The subsequent measurements were conducted for the dye eosin-Y in ethanol. The average LIF and Mie scattering signals of single droplets are shown for a range of about 25 μ m to 60 μ m. Some example images are presented in Figure 3.

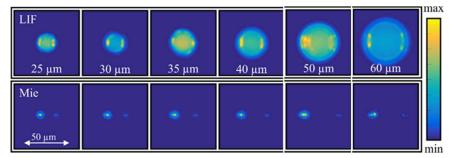


Figure 3: Averaged LIF and Mie scattering signals from micro-droplets in diameter ranging from 25 μ m to 60 μ m. The intensity of each image is normalized to their individual maximum value. The light sheet enters the droplet from the right.

It is obvious that the LIF signal is not homogeneously produced in the whole droplet. The signal is stronger in the center of the droplets due to the larger illuminated and fluorescing volume. However, some droplets show also clear signs of MDR with bright spots in the signal and rings at the droplet surface, which is addressed below.

The Mie images consist exclusively of two glare points at the left and right side of the droplets (not shown here).

The Mie-scattering of individual droplets is very sensitive to the angle of detection. Furthermore, it depends on the droplet size and does actually not follow a potential trend. A calculation of the Mie scattering intensity show strong intensity oscillations with droplet size. However, the chosen detection angle of the camera dampens signal oscillations and allows the usage of a potential fit function. Thus, a dye concentration of 0.5 vol % eosin-Y in ethanol showed a good agreement with the d^3 - (LIF) and d^2 - relation (Mie), respectively.

The LIF calibration function can be described by the following equation (power law) at standard conditions:

$$I_{LIF} = 43.94 \cdot d_{droplet}^{3.33} \,. \tag{3}$$

An exponent greater than three occurs, which is mainly attributed to the MDR emission in droplets (Le Gal et al. 1999).

The fit curve of the Mie signal can be described with the following equation:

$$I_{Mie} = f(d_{droplet}) = 7062.5 \cdot d_{droplet}^{2.12}$$
(4)

The Mie-signal calibration curve shows a good agreement with the d^2 -dependency. However, an exponent greater than two is present, which was also reported by Le Gal et al. (Le Gal et al. 1999), and which again has been explained by occurrence of MDRs.

The corresponding LIF/Mie ratio is described by the following dependency:

$$I_{\underline{LIF}} = f(d_{droplet}) = 0.0054 \cdot d_{droplet}^{1.25}$$
(5)

This calibration curve roughly exhibits the d-dependence according to the hypothesis of the LIF/Mie droplet sizing approach (Le Gal et al. 1999, Domann und Hardalupas 2003). The standard deviation is not shown in this diagram, however, the 1- σ uncertainties are in the range of 5.5 % for a droplet size of 30 μ m.

The effects of dye concentration are investigated at 0.25 vol %, 0.5 vol % and 0.75 vol % at constant laser energy and fuel temperature (293 K). In practical applications for spray measurements using PDS, the dye concentration in the fuel should be large enough in order to guarantee a sufficiently large signal. However, a variation of the dye concentration would lead to distinct errors in droplet sizing.

Figure 4 exhibits that an increase of the dye concentration leads to a proportional increase in the LIF/Mie ratio. This trend is expected from the LIF-signal dependency on the dye concentration for the linear fluorescence regime. Additionally, an increased dye concentration may lead to a slightly larger light extinction within the droplets. This could again result in a decreased Mie signal. In general, a dye enrichment could occur in case of distinct fuel evaporation, e.g. under increased ambient temperature especially for small droplets (Koegl et al. 2020). In ref. (Koegl et al. 2020) the effect of droplet evaporation on the dye enrichment was studied numerically. For single isolated droplets at a velocity of 20 m/s (which is reasonable for DISI sprays), after 1.2 ms and pure ethanol, a small droplet of initially 5 μ m diameter would show a dye concentration increase of 46.3%. A droplet size of 10 μ m would result in a dye concentration increase of 13.0% (20 μ m: 3.1 %; 40 μ m: 3.1 %). However, this would be a worst-case scenario since in a spray the evaporation is limited due to the vapor saturation of the ambience due to evaporation of neighbouring droplets.

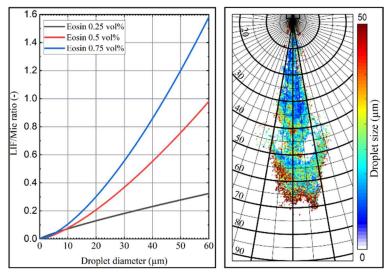


Figure 4: Calibration curves of the LIF/Mie ratio at various dye concentrations of eosin-Y in ethanol (left) and SMD measurements in a DISI spray (0.2 MPa chamber pressure, 16 MPa injection pressure, 293 K fuel and ambient temperature, right) for ethanol doped with eosin-Y.

Another important quantity determining the accuracy of PDS is the droplet temperature itself, which may change during the injection process due to droplet heating or cooling. The effects of liquid temperature were studied at constant laser energy and dye concentration (0.5 vol. %). The data can be found elsewhere (Koegl et al. 2018) and only a summary is given here. The LIF/Mie ratio was studied for three temperatures between 253 K and 333 K. In principle, the LIF/Mie ratio increases with an increase in fuel temperature. Between 253 K to 293 K, the LIF/Mie ratio increases by about 15 %, e.g. for a droplet size of 30 μ m. This increased LIF-signal can be explained by the larger absorption cross section at increased temperatures. An increased absorption in the droplet would result in smaller Mie-scattering intensity resulting in a larger LIF/Mie ratio. However, the increase in LIF/Mie ratio is about 40 % between 293 K and 333 K for a 30 μ m large droplet. This is probably because of the additionally occurring partial evaporation of the fuel leading to a dye enrichment. This would result in a larger LIF, but lower Mie signal, and again to a much higher LIF/Mie ratio. In general, increased uncertainties up to 29 % in droplet size may occur for large droplet temperature variations of 40 K and the resulting dye enrichment due to evaporation.

Finally, the LIF/Mie calibration curve at a dye concentration of 0.5 vol% was applied to extract the droplet Sauter Mean Diameter (SMD) in a transient ethanol spray that was studied in an injection chamber. The injection duration was 1.8 ms. This droplet size mapping was done using Structured Laser Illumination Planar Imaging (SLIPI) in order to suppress the artifacts induced by multiple light scattering. A two-pulse SLIPI technique was applied as described in reference (Storch et al. 2016). Figure 4 (right) presents calibrated single-shot SLIPI-LIF/Mie ratios in terms of droplet SMD in the central plane of one jet of the multi-stream DISI spray at a point in time of 2.55 ms aVSOI (after visible start of injection). The injector tip is in the top of the image. Large vortices are visible in the left and right spray boundary. The droplets in the near nozzle region (0 mm to 10 mm) should not be considered due to probable existence of non-spherical droplets and ligaments in that regions. This means that the calculated SMD values may not be realistic in that region. The large droplets at the lateral spray edges are in the range of 25 μ m - 35 μ m. The large droplets at the spray front are 50 μ m and larger. This is mainly due to droplet coalescence mechanisms. In general, it could be shown that the PDS technique provides reasonable and very promising results of the spray structure in terms of SMD in single shot measurements.

In the subsequent section, another dye is tested for studying fuels with lower polarity including realistic gasoline or aviation fuels and their surrogates such as alkanes. This dye selection is required as eosin-Y is not dissolvable in such fuels or otherwise a significantly increased amount of a polar fuel component must be added.

Calibration data based on Nile red for non-polar solvents

The subsequent measurements were conducted for the base fuel "Toliso", which is a substitute fuel for gasoline or kerosene. It consists of 35 vol.% toluene and 65 vol.% isooctane. Additionally, an ethanol admixture of 20 vol.% was investigated in order to study effects on the LIF and Mie signals. The measurements were conducted for a dye concentration of 100 mg/L. In total, more than 30,000 single droplets were analyzed for both fuels. The LIF and Mie-scattering signals of the droplets show similar characteristics like the ethanol droplets doped with eosin-Y. The signals in Figure 5 are normalized to the individual maximum in the respective image.

Toliso Mie 30 μm 35 μm 40 μm 45 μm 50 μm 25 μm 30 μm 35 μm 40 μm 45 μm 50 μm E20 LIF 30 μm 35 μm 40 μm 45 μm 50 μm 25 μm 30 μm 35 μm 40 μm 45 μm 50 μm E20 Mie 40 μm 40 μm 45 μm 50 μm	Toliso LIF 25 μm	О 30 µm	О 35 µm	40 µm	45 µm	50 µm
25 μm 30 μm 35 μm 40 μm 45 μm 50 μm	. • •	3 0 μm	35 μm	4 0 µm	45 μm	50 μm
E20 Mie	•	О 30 µm	<mark>35 µm</mark>	40 µm	45 μm	50 µm
25 μm 30 μm 35 μm 40 μm 45 μm 50 μm	25 μm	30 μm	3 5 μm	40 µm	45 μm	50 μm

Figure 5: Bin-wise averaged LIF and Mie scattering droplet data of toliso and E20 (20 vol.% ethanol in toliso), 293 K, nile red 100 mg/L). Extracted from (Koegl et al. 2020).

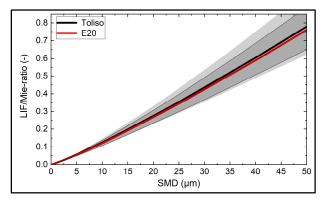


Figure 6: Calibration curves as a function of droplet size for Toliso and E20 based on the dye nile-red. Data below 20 μm are extrapolated.

The LIF/Mie ratios of toliso and E20 at 293 K given in Figure 6 can be described by the following equations:

$$I_{LIF/Mie_Toliso} = f(d_{droplet}) = 0.0092 \cdot d_{droplet}^{1.14}$$

$$I_{LIF/Mie_E20} = f(d_{droplet}) = 0.0087 \cdot d_{droplet}^{1.14}$$
(5)
(6)

The fit curves show a similar trend for both fuels under investigation. The resulting microscopic LIF/Mieratio exponents are not dependent on the ethanol admixture. The value of exponent is identical (1.14) and there are slightly different pre-exponential factors. However, the uncertainty is slightly larger for toliso compared to E20, which is, for example, \pm 20% for toliso and \pm 16% for E20 at 30 µm. This could be explained by different absorption and emission behavior of the dye in the respective fuels, which also affects MDR emissions.

For clarifying the MDR effects, a spectral characterization of the fluorescence emission was conducted in the droplet chain for various fuel mixtures, see Figure 7. It should be noted that iso-octane was used as base fuel instead of toliso. However, the trends should be comparable. For this purpose, a fibercoupled spectrometer (Model USB 4000, Ocean Optics, USA) was applied.

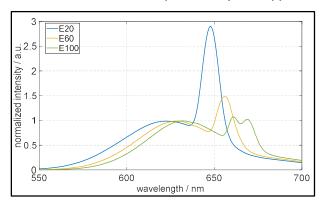


Figure 7: Spectral LIF intensities for 100 µm isooctane/ethanol droplets; signals are normalized to the initial fluorescence peak; all fuel mixtures contain 100 mg/l nile red, conditions: 293 K, 0.1 MPa.

Again, a blueshift of the fluorescence band of nile red was observed. This is similar to the measurements in the cuvette shown above. Additionally, the presence of MDRs is visible through distinct peaks. These peaks occur at larger wavelengths than the natural fluorescence peak of the fuel/dye mixture. The MDR peak is strongly dependent on solvent and droplet size (droplet diameter effects are not shown here). Larger ethanol concentration lead to a reduction of the MDR peaks.

The decreasing MDR signals in the LIF images for larger ethanol concentrations were also observed in droplet images (see Koegl et al. (Koegl et al. 2021)). There, larger values of the COV (coefficient of

variation) in the LIF signals were visible for lower ethanol concentrations. Consequently, in future work, the MDR effects in the calibration data need to be compensated either by selection of appropriate filters or by adding another dye for reabsorption of the lasing peaks. The latter is part of current work for 2-color thermometry using a dye couple. Other effects affecting the accuracy of the PDS technique such as temperature variations and dye enrichment during evaporation must be addressed as well.

Conclusions and future work

This paper reviewed the current state of the PDS-technique for droplet sizing in ethanol and gasolineor kerosene-like fuels based on the LIF/Mie signal intensity ratio. The studied fuels were surrogate fuels isooctane and isooctane-toluene mixtures, as well as biofuels such as ethanol. The admixed dyes are eosin-Y and nile red, which are soluble either in polar or non-polar fuels. The effects of laser irradiation, dye concentration, and temperature on the calibration curve were discussed in a wide range of droplet sizes. The precision of the calibration was calculated to be 5.5 % for a droplet size of 30 µm. A major source of error is the dye enrichment for conditions with strong fuel evaporation, which is an issue for smaller droplets. For example, simulations of single isolated evaporating droplets with 5 µm initial droplet size showed an increase in dye concentration of 46 % within 1.2 ms, which would lead to a proportional overestimation in droplet diameter. However, this would be a worst-case scenario since the evaporation rate in sprays is usually much lower. The calibration data was utilized for SMD measurements in an ethanol spray produced by a DISI injector. In dense sprays, additional error sources are present. For this purpose, structured laser illumination planar imaging (SLIPI) was applied in order to suppress the artifacts induced by multiple light scattering in denser spray regions. Very promising SMD measurements on a single shot basis were possible under realistic conditions that are relevant for IC engine and many other technical sprays.

Calibration data for nile red in surrogate fuels "toliso" and E20 showed very similar LIF/Mie ratio curves. Larger uncertainties were observed for toliso, which could be explained by different absorption and emission behavior of the dye. For both dye concepts, effects of "lasing" or "morphology-dependent resonances" (MDR) were observed in the LIF images. Furthermore, dye enrichment and temperature variations during evaporation may cause large errors in the LIF/Mie-ratio, which are part of present and future work.

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