

# Spectral and angular analysis of non-linear optical response of soot exposed to femtosecond laser light

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## Abstract

The present work shows an application of non-linear optics (NLO) with the aim to characterise aerosol particles by the measurement of the spectral response of their radiated light at 0° and 30° when exposed to a femtosecond laser light beam. A first optical setup has been developed to carry such measurements while ensuring to avoid any laser/optics pollution in the results. While Second Harmonic Generation (SHG) or Third Harmonic Generation (THG) were difficult to observe, a strong spectral signature of other NLO phenomena, such as fluorescence induced by femtosecond induced filamentation (FLIF) are observed. These signals are analysed in terms of their amplitude for different aerosols (CAST soot particles and DEHS particles). The results suggest, that weak SHG signals might exist but that they are covered by FLIF. Finally based on these findings, a novel experimental setup for future works is proposed.

## Introduction

Black carbon (BC) nanoparticles, also known as soot, are formed during the incomplete combustion of hydrocarbons. BC particles are known to react with molecules in the atmosphere, causing changes in their radiative properties, which can lead to alterations in the heat transfer balance and have been identified as a significant contributor to global warming [1, 2]. The morphological and compositional complexity and variability of BC particles require a detailed characterization to facilitate the understanding of their formation and the validation of theoretical models related to their formation, aging processes, and climate impact. Additionally, the toxicity of BC particles is strongly influenced by their internal structure, morphology, and composition, and they can cause oxidative stress or inflammatory responses upon exposure on human cells [3]. The fractal morphology of BC particles can also increase the reaction rate between Polycyclic Aromatic Hydrocarbons (PAH) and human cells, which has implications for their impacts on human health [1, 2].

Numerous diagnostics based on the particles' optical and radiative properties exist giving access to measurands as volume fraction, particle size, morphology, fine structure, and elemental and molecular composition. Classic ex-situ methods, such as Transmission Electron Microscopy (TEM) [4, 5], Mobility Particle Sizer Spectrometer (SMPS) [6], Tapered Element Oscillating Microbalance (TEOM) [6] or Raman spectroscopy [7], require the particles to be probed, thus altering their physical properties [8] and excluding the possibility to obtain spatially or temporally resolved measurements. Due to these limitations, numerous in-situ methods, mostly based on the interaction between the particles and laser light, have been developed [9]. Common techniques are Line-of-Sight Attenuation (LOSA)

[10], Laser Induced Incandescence (LII) [11], Angular and spectral light scattering [12], Small Angle X-ray Scattering (SAXS) [13] or Time Resolved Fluorescence Anisotropy (TRFA) [14] which all give access to the particle concentration, size and morphology. Moreover, Laser induced fluorescence (LIF) can be used to detect some PAH compounds but it is difficult to be quantitative and to separate the contribution of gas and particle phases [15]. While all these techniques give valuable information on the aggregate scale, no mature in-situ technique is currently available, which would allow to examine the particles' internal structure or chemical composition.

Non-Linear Optics (NLO) is a promising approach to gain access to these properties of black carbon (BC) particles. NLO describes the optical response of a medium to high intensity/femtosecond laser light, which can exhibit various multi-photon phenomena [16]. Second Harmonic Generation and Third Harmonic Generation (SHG and THG) are effects observed when molecules are exposed to such light sources, corresponding to the generation of light with half and one third of the incident light wavelength, respectively. The existence of SHG and THG is dependent on the internal structure of the medium. In the case of BC particles, which are constituted of crystalline, amorphous, and organic compounds with different symmetries, NLO effects could be applied to distinguish the different constituents. This would provide valuable information on the internal structure and chemical composition of the BC particles [16]. NLO is thus a promising new approach that may provide additional insights into the composition and structure of BC particles.

The utilization of Second Harmonic Generation (SHG) and Third Harmonic Generation (THG) for internal structure analysis is not new and has been implemented in other scientific domains such as biology [17] and chemistry [18], where non-linear microscopy

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is used to analyze solid or liquid probes. However, the application of this technique to aerosol phase particles such as BC has been limited. In a recent study, SHG was detected on NaCl seeded, 100nm diameter, water aerosol particles, coated with DiA4 or crystal violet (both known to produce resonant enhancement of SHG) [19, 20], but this approach may potentially alter the particles' inner structure. Additionally, another study observed THG and scattering on soot particles produced in flames [21]. As such, the structure of BC particles and other aerosols remains largely unexplored with these techniques and a large variety of questions thus remain open. Moreover, to the authors' knowledge, no measurement of SHG has yet been carried out on BC particles in-situ in aerosol phase.

Some works on SHG took an interest in the measurement the effect of particle size on the angular scattering profile of polystyrene spheres in water (with radius between 50nm and 200nm) interaction with high intensity laser light (800nm and 80fs pulses) [22]. This angular signature is also observe from numerical simulations using analytical models [23].

In addition, other effects such as femtosecond laser induced filamentation (FLIF) [24] and/or the excitation of the aerosol particles or combustion intermediates by a filament as it can be seen with Raman measurement[25, 26] or filamentation induced fluorescence [27, 28] cannot be excluded due to the energetic light pulses from the laser. This laser induced filamentation is known to lead to fluorescence emissions showing a specific and strong spectral signature [29]. However, for now this type of signals has only been observed in flames and not yet applied to soot particles alone. The comparison of these signals with or without flame could give information on the soot contribution in their generation.

In the last 2 years, the present works' authors have developed an experimental setup for analyzing the interaction of aerosols including soot particles in a particle flow or in a flame with a femtosecond laser light beam, by measuring the temporal response at 0° and 30° as well as the spectral response at 30° [30]. Based on existing studies on the NLO response in flames [21], aerosols [19, 20] and electric fields [31], an optical setup was developed and optimized to measure weak SHG signals in a reliable way. The setup is used to carry out measurements on aerosol soot particles generated by a Mini-CAST, whose particle properties are characterized in the literature [6], and on DEHS aerosol particles. This paper presents some results from this experimentation and a new experimental setup that gives access to the angular, spectral and temporal NLO signals generated by femtosecond laser light interaction with BC aerosol particles.

### Experimental setup

The current optical setup, shown in Fig.1a aims at focusing the high-intensity laser beam onto the measurement volume and collecting the NLO signals result-

ing from the laser/particles interaction. A femtosecond laser system Ti-Sapphire amplifier (Coherent Libra) at 800nm with 1kHz repetition rate, pulse energy of 3.5mJ and pulse duration of 120fs is used as the laser source. A first polarizer (Pola1) is rotated to control the power range sent to the following optics. The beam then passes through a Half waveplate and polarizer assembly (HW1 & Pola2) for which the halfwave plate is rotated to fine-tune the incident laser power on the sample. Pola2 is followed by a second half-wave plate (HW2) which fixes the polarization state of the laser light, and then by an 800nm bandpass filter (20nm bandwidth – Filter1) to avoid SHG from the light-matter interaction in the half-wave plates, as observed in previous works [32].

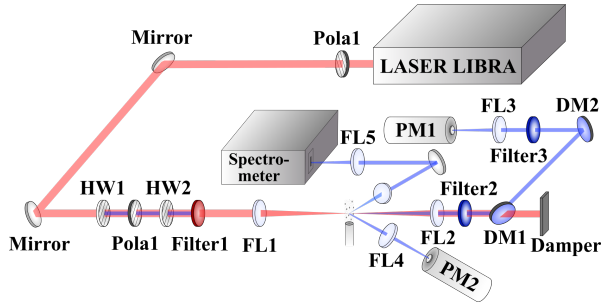
The beam is then focused into a region where soot particles are generated using a convex lens of 300mm focal length. The scattered light from this region is collected by three different detection lines:

-1. The first line has the aim to temporally analyse the intensity of the NLO signal at 0°. The signal is collected and collimated using a convex lens of 200mm focal length, followed by a 150nm bandwidth bandpass filter centered at 400nm (Filter2). Filter2 absorbs a significant proportion of laser pump light, ensuring that no SHG is generated in the following optical components. The remaining light is then reflected by two dichroic mirrors (DM1 and DM2), further separating the remaining laser pump light from the signal. Finally, a 10nm bandwidth optical filter (Filter3) centered at 400nm is placed in front of the Photomultiplier (Hamamatsu - H3378-50) on which the signal is focused by a 150mm focal length lens. The use of DM1, DM2, and Filter3 ensures that no 800nm laser light arrives at the detection, while Filter1 and Filter2 ensure that the setup measures exclusively the SHG generated in the measurement volume.

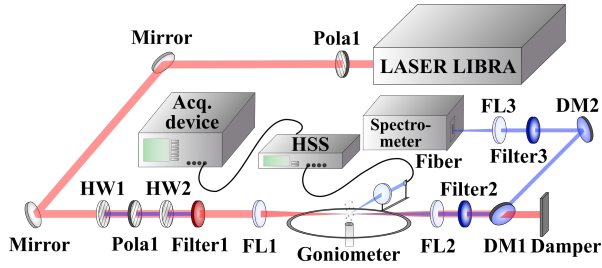
-2. The second detection line aims at measuring a temporally resolved NLO signal at different intensities at 30 degrees. For this purpose, a second photomultiplier (Hamamatsu - H3378-50) is used after focalising the signal with another 150mm focal lens. No dichroic mirrors are used (contrarily to the 0 degree line where the 800nm pump light can damage the following optical components), but a 800nm to filter the elastic scattering of the pump light.

-3. Finally the third detection line aims at measuring the spectral signature of the NLO signal at 30 degrees. As depicted in Fig.1a, the scattered light is collected at 30° using a 150mm focal lens and focused in a Princeton Instrument Acton SpectraPro SP2150 spectrometer (grating : 300g/mm) coupled with a PIMAX4-MT camera. The spectral observation is essential to determine the nature of the signal, for example whether it is due to NLO phenomena such as harmonic generation or from fluorescence generated within the laser filamentation.

The results presented in the following section were carried out with the current setup, as depicted in Fig.1a. As a consequence from the presented results, an enhanced experimental setup is being worked on, as pre-



(a) Current optical setup for the measurement of NLO responses of aerosols at 0° and 30°.



(b) Enhanced optical setup for spectrally, temporally and angular resolved measurement of NLO responses of aerosols.

Figure 1: Optical setups for the measurement of the optical response of the interaction of aerosol streams with a femtosecond laser light beam.

sented in Fig. 1b, with the capability of determining a spectrally, temporally and angular resolved signature of the NLO response of the analysed particles, thus allowing to identify the nature of the scattered signals and put into evidence their dependence on soot particle structure and composition.

The future experimental setup is similar to the current one, except for the detection system. The photomultiplier used to measure the signal at 0° is switched for the spectrometer previously used for the detection at 30° in the previous experimental setup described in this section. Moreover, the angular detection will be carried out by using two hamamatsu fibered multianode High Sensitive compact Spectrometers (HSS), alternately used for the UVs and visible range, which will be mounted on a motor controlled rotation system.

These optical setups allow to measure the optical response of the interaction between a jet of soot particles generated by a MiniCAST as well as on DEHS aerosol particles or any other types of aerosols. Measurements could also be conducted over bulk solution or in flames without the need to fundamentally modify the optical part of the setup.

## Results

The different temporal and spectral measurements were taken at different pump laser intensities. In general, the response intensity is increasing with increasing pump power, following a power law. The detected signal is extremely weak and seems related to fluorescence generated by FLIF resulting from the local modi-

fications of the optical index (Kerr effect), like observed in remote filament-induced breakdown spectroscopy (R-FIBS) [33]. The most notable results will be shown and discussed in the following.

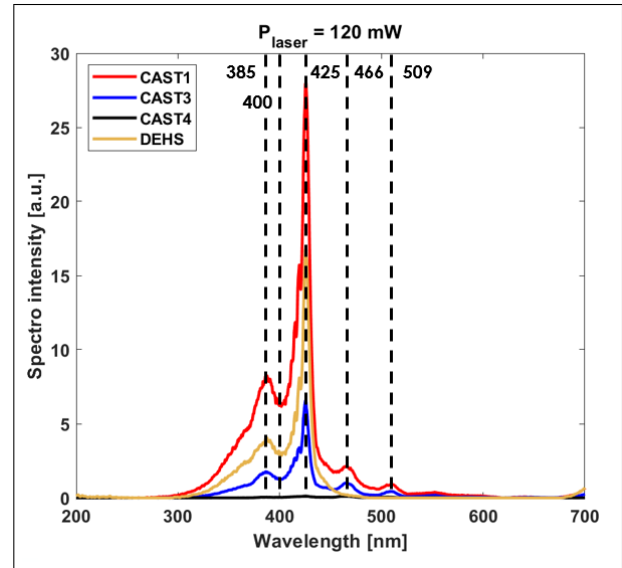


Figure 2: Spectral responses of the scattered light at 30° of CAST and DEHS aerosol particles with a femtosecond laser light beam at an average power of 120mW.

Figure 2 shows the spectrometer measurement at 30° (see Fig. 1a) for the aerosols of CAST1, CAST3, CAST4 [6] and DEHS particles due to their excitation with the femtosecond laser at 800nm at a power of 120mW. Several distinct peaks are visible at 385nm, 425nm, 466nm and 509nm with a width from 20 to 30nm. No peak is observable at 400nm, where the SHG signal is expected. Even if the signal could be dependent on the particle density, it seems to show the same behaviour with different intensity depending on the aerosol characteristics, with the highest peak amplitudes for CAST1 followed by DEHS and CAST3 and finally the CAST4 that presents a very weak signal. It is also remarkable that the peaks at 466nm and 509nm seem only to appear for soot aggregates.

The strongest peak can be observed at 425nm and 385nm and could correspond to the transitions emissions of CH at 430nm and cyano radical CN at 388nm, respectfully presented in combustion intermediates sensing by femtosecond filament excitation [27, 29]. However this work present results for alcohol flames and the CAST is a device generating particles from a propane/nitrogen/air flame. Moreover, the burnt gas concentration in the particle flow from the CAST is considered negligible. The existence of these peaks for DEHS is however remarkable, since, according to [27] the peaks are related to combustion species and no burnt gases do exist in the DEHS particle stream. The width of these peaks goes from 20 to 30nm which is comparable to the results of the previously cited results [27, 29] or could come from the spectrometer resolution.

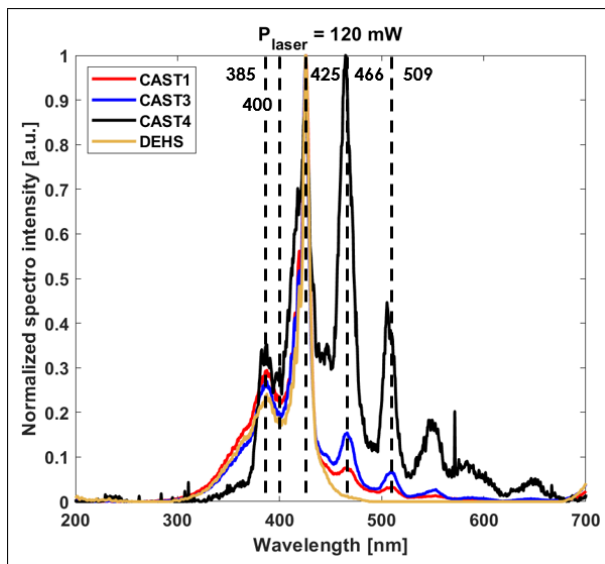


Figure 3: Spectral responses of the scattered light at 30° of CAST and DEHS aerosol particles with a femtosecond laser light beam at an average power of 120mW, normalized by their intensity at 425nm.

Due to the difference in intensity, the signal from the CAST4 conditions appears to be too weak to be measured. Thus, Fig.3 shows the same signals normalized by their own maximum in order to put forward the different peak structures.

The peaks visible at 385nm and 425nm present the same behaviour in proportion to each other and seem common no matter the nature of the aerosol is. Nonetheless, the peaks visible at 466nm and 509nm seems to be dependant to the aerosol nature, as their intensity seems more important in comparison to the peak at 425nm for the CAST4 conditions, visible at lower intensity for CAST1 and CAST3 and not detected for the DEHS particles.

For the CAST4 particles, a weak peak could also be observed at 400nm. That could mean that SHG signal exists from this type of laser particles interaction but in very small quantities compared to the other FLIF signals that are detected in most cases. A possible reason could be that fact that the FLIF is sufficiently weak for CAST4 (contrarily to the other cases), that it does not cover possible, orders of magnitude weaker, SHG signals.

Fig.4 shows the evolution of the intensity of the signal at 400nm normalized by the scattered intensity at 425nm. It also shows their least-square fit by a power law  $y = ax^b$ . The CAST1, CAST3, DEHS and Air signals increase weakly, while the signal at 400nm for the CAST4 particles seems close to quadratic. This could suggest that the 400nm peak is associated to SHG, however more evidence would be necessary.

## Conclusions

Understanding the internal structure, morphology, and composition of BC particles is essential for developing theoretical models related to their formation, ag-

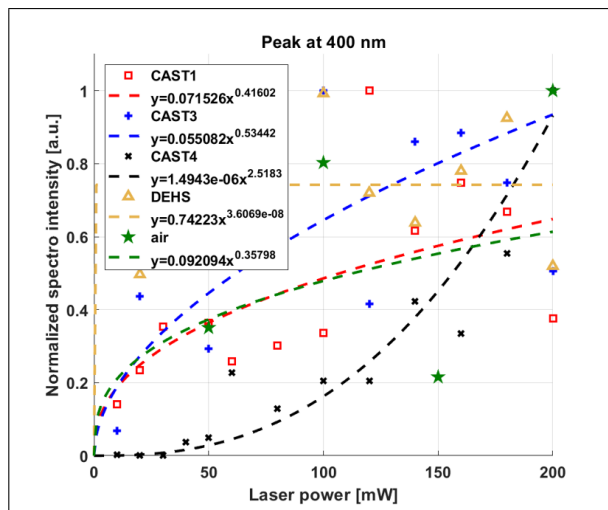


Figure 4: Intensity of the emitted light at 30° and 400nm of CAST and DEHS aerosol particles flows and air exposed to a femtosecond laser light beam, within the laser average power, normalized by the intensity of the 800nm elastic scattering and the intensity at 425nm.

ing processes, and climate impact. While various ex-situ and in-situ methods exist for measuring the properties of aerosol particles, non-linear optics (NLO) is a promising new approach that may provide additional insights into the composition and structure of BC particles. For testing the potential of NLO for BC analysis, a novel optical setup has been designed in order to measure the scattered NLO response from aerosol jets exposed to a femtosecond laser light beam. Scattered signals have been observed at an angle of 30° and analysed by a spectrometer. The spectra show some peaks that could be induced by fluorescence due to the filamentation of the laser beam [27], as other peaks seem specific to the nature of the aerosol particles and more specifically to soot characteristics. Typical NLO effects as Second Harmonic Generation (SHG) seem complicated to observe due to their weak intensity in comparison with these other NLO signals while using high intensity femtosecond laser light.

However, these particles/filamentation interactions show signatures associated with the different types of soot particles and might in the future give access to information on their properties. THG is still to be observed in the UV range. Although the application of this technique on aerosol phase particles such as BC is still in its early stages, it holds great potential for advancing our understanding of the properties and behavior of these important atmospheric pollutants. It is for this purpose that the experimental setup showed in Fig.1b is proposed, allowing to extend the investigation on the nature and behaviour of these signal on a temporal, spectral and angular aspect. By conducting these measurements for different types of aerosol particles, the relation between FLIS and SHG/THG at different angles will be tested and their suitability for developing

a new class of in-situ soot diagnostics will be examined in future works.

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