Polarization effects in Raman spectroscopy of light-absorbing carbon

Kim Cuong Le | Jonatan Henriksson | Per-Erik Bengtsson

Abstract

Raman spectroscopy is widely used for characterization of carbon-based materials including light-absorbing carbonaceous (LAC) matter. However, information on polarization properties of these materials are generally lacking, and often the polarization characteristics of experiments are not presented in the literature thereby giving uncertainty to presented data. In this work, the polarization properties of various LACs are studied using Raman spectroscopy. The Raman spectra have been compared while varying the relative polarization, particularly the relative direction of the electric field between the incident light and the detected scattered light. Specifically, the intensity ratio between the $D_1$ and $G$ peaks ($I_{D_1}/I_G$) was analyzed in parallel and perpendicular polarization configurations for samples taken from a sooting flame, in the exhaust of a soot generator and carbon blacks. In this work, the parallel and perpendicular polarization configurations mean that the polarization of the detected scattered light is parallel and orthogonal, respectively, to the linearly polarized incident light. It was found that the ($I_{D_1}/I_G$) ratio decreased in the more ordered carbonaceous structures when changing the polarization configuration from parallel to perpendicular. Additionally, based on the depolarization ratios ($D_p < 0.75$), the prominent Raman peaks of LAC were found to originate from totally symmetric vibrations. For the $G$ peak, the depolarization ratio was measured to be in the range 0.46–0.56 for all LAC materials.

KEYWORDS

carbon black, light absorbing carbon (LAC), polarization, soot

1 INTRODUCTION

Light-absorbing carbonaceous (LAC) matter is defined as light-absorbing, carbonaceous substances emitted by incomplete combustion of organic compounds. Their existences in the atmosphere and on the Earth surface contribute to the global warming. In addition, there are numerous epidemiological studies that have presented established associations between exposure to particulate pollution, including LAC, and increased morbidity and mortality for respiratory, cardiovascular neurasthenia, asthma, and obstructive pulmonary diseases.

Because of the harmful consequences of LAC, a variety of techniques have been developed to detect and study LAC. Among these techniques, Raman spectroscopy has been used since decades due to its...
nondestructive properties and its high sensitivity in characterizing the nanostructure of carbonaceous materials including LAC. Analysis of positions and widths of characteristic Raman peaks, as well as of ratios between different peaks, has been used to identify the internal bonding structure of LAC.

Raman investigations of disordered carbonaceous materials such as LAC presented in the literature often lack information on polarization effects and could potentially be a source of uncertainty in spectral interpretation. Indeed, polarization studies using Raman spectroscopy have previously been made on highly ordered carbonaceous materials such as graphene, graphite, and carbon nanotubes (CNTs). Most of the studies on graphene focused on polarization anisotropy of the double-resonant $2D_1$ band and the graphic $G$ band. The polarization dependence is a direct consequence of inhomogeneous optical absorption and emission mediated by electron–phonon interaction in Raman scattering process. This thus makes polarized Raman spectroscopy become one of the important tools in studying low-dimensional crystal materials. However, with disordered structures of LAC, their polarization behavior is expected to be different.

In this work, six different LAC materials were investigated, including flame soot, freshly emitted soot from a soot generator, and carbon blacks. The polarization properties of the Raman peaks in both the first-order and the second-order spectra were studied. When applying different linear polarizations of incident and detected scattered Raman light, relative intensity differences could be observed between the different LAC materials.

## Samples and Methods

Six LAC samples with a large variation in nanostructures and composition were investigated in this work. Two samples of freshly emitted soot produced by a mini-CAST 5201C soot generator were selected with characteristics similar to black carbon (OP1) and brown carbon (OP6). Specifically, OP6 soot contains nearly 60% of organic and pyrolytic carbon per total carbon, while this fraction is less than 10% for OP1 soot. Two other soot samples were collected from a premixed flat ethylene/air flame at an equivalence ratio $\Phi = 2.3$ at 8- and 18-mm height above the burner (HAB). As incipient soot forms at a height of 4 mm above the burner and grows for increasing flame height, the sample at 8 mm represents younger more immature soot with lower absorption efficiency, whereas the 18-mm sample consists of more mature soot with higher absorption efficiency. The increase of maturity mentioned in this work refers to the more ordered and more graphitized structures. In addition, we used two carbon black products of Norcarb Engineered Carbons AB, named INR-A and CXN-550, where the INR-A sample was a heat-treated standardized carbon black. Via iodine absorption analysis, the iodine number of CXN-550 is higher than that of INR-A indicating a larger amount of unsaturated substances in CXN-550.

As Raman signals from substrates where the samples are deposited could interfere with the Raman signals from the samples, a thickness of few hundred micrometers was required to eliminate such interference. During the sampling process, sapphire windows were placed at the exit of the exhausted pipe of the mini-CAST soot generator to collect the flame by-products. It took 30 min for OP1 sample and 2 h for OP6 sample, to deposit sufficiently thick layers on the windows for the subsequent Raman analysis. With the flame soot, at each specific height, the sapphire windows were repeatedly moved in and out the ethylene/air flame many times until a sufficiently thick soot layer was formed. This frequent repetition and few seconds sampling each time had the purpose to keep the window at sufficiently low temperature to avoid oxidation of the sampled soot when kept outside the flame. It took 5 min and 2 h for sampling the flame soot at HAB = 18 mm and HAB = 8 mm, respectively.

All Raman studies on disordered carbonaceous materials, reported to date, were performed in the backscattering configuration. Similarly, in this work, Raman spectra were obtained at room temperature under atmospheric pressure using a homebuilt backscattering Raman setup, which is described in detail previously. Briefly, we excited the samples by a diode laser at 532 nm and with a power density on the samples of around $6 \times 10^3$ mW/mm$^2$. The scattered photons were focused on an optical fiber, after being filtered by a long-pass edge filter (Thorlabs, FEL0550) transmitting above 550 nm and a notch filter (Thorlabs, NF533-17) for total suppression of the laser wavelength. The obtained signals were detected by an IsoPlane SCT320 spectrograph connected to a charge-coupled device (CCD) camera (Andor technologies, Newton DU94ON-BV). All spectra have the same acquisition time of about 10 min. We used a He–Ne lamp for wavelength calibration. In addition, all spectra were corrected with a calibration curve for the detection system. This curve is the fraction between the standard spectrum of an unpolarized white light source and its experimental spectrum obtained by our instrument.
For the polarization experiments, the incident light was linearly polarized, and relative polarization between the incident light and the detected signals could be precisely controlled by rotating a polarizer mounted in front of the optical fiber. In this work, we use linearly polarized incident laser light on the samples. For the parallel (p) polarization configuration discussed in this work, the polarization axis of the polarizer is parallel with the electric field vector of the incident light, whereas for the perpendicular (s) configuration, the polarization axis of the polarizer is orthogonal to the electric field vector of the incident light.

3 | RESULTS AND DISCUSSION

3.1 | Spectral characteristics

Figure 1 shows Raman spectra, including the first (800–1,700 cm$^{-1}$) and the second (2,300–3,500 cm$^{-1}$) orders of the six samples as a function of Raman shift. These spectra were normalized with the $G$ peak intensity (considered from the top to the bottom of $G$ peak) and vertically translated on top of each other. The typical characteristics of the first order in all Raman spectra are two prominent peaks, called $G$ ($\omega_G = 1,580$ cm$^{-1}$) and $D_1$ ($\omega_{D1} = 1,355$ cm$^{-1}$). The $G$ peak, which is assigned to the C=C stretching vibration mode, is observed in either aromatic rings of graphitic structures or olefinic chains.$^{[23]}$ The $D_1$ peak, so-called disorder mode and activated by defects, indicates the $A_{1g}$-type mode at the $K$ point of the Brillouin zone.$^{[23]}$ For some of the cases, the Raman spectra rise above a strong fluorescence contribution, which is due to organics such as polycyclic aromatic hydrocarbons (PAHs) in the samples.$^{[6,8]}$ This is especially evident for the mini-CAST OP6 sample. With low fluorescence signals, it can be fitted using a second-order polynomial function and subsequently subtracted from the total spectrum before Raman analysis was applied.$^{[8]}$ With the strong fluorescence contamination in the Raman spectrum of the OP6 soot, we deposited and measured the organic compounds vaporized from heat-treated OP6 sample at 500°C.

Besides stronger fluorescence contamination, the more disordered and less mature samples, such as the mini CAST soot, the flame soot at HAB = 8 mm, and CXN-550 carbon black, also show broader peak widths. This is due to the appearance of many additional peaks to the $D_1$ and $G$ peaks, such as $D_4$ ($\omega_{D4} = 1,180$ cm$^{-1}$), $D'_1$ ($\omega_{D'1} = 1,270$ cm$^{-1}$), $D_3$ ($\omega_{D3} = 1,500$ cm$^{-1}$), $D_2$ ($\omega_{D2} = 1,620$ cm$^{-1}$), C–H “out of plane” peak ($\omega_{C-H} = 850$ cm$^{-1}$), and peaks assigned to oxygenated hydrocarbon ($\omega_{ether} = 1,060$ cm$^{-1}$) or $\omega_{carbonyl} = 1,750$ cm$^{-1}$), and sp hybridized bands ($\omega \sim 1,800–2,100$ cm$^{-1}$). These peaks are due to complex structures with the existence of polyaromatic subunits, mixture (linear, trigonal, and tetrahedral) hybridization states, amorphous carbon, oxygenated hydrocarbon species, unsaturated substances, and organic compounds beside of dominating graphite-like structures. The detailed information about their origins was reviewed in our recent paper.$^{[6]}$

The presented Raman spectra also contain the second-order bands in which two pronounced peaks at $\sim$2,700 and 2,900 cm$^{-1}$ have been assigned to the $2D_1$ overtone and $D_1 + G$ combination bands. The $2D_1$ mode is an overtone of the $D_1$ peak where the electron is back-scattered by a second phonon instead of a defect.$^{[24]}$ This peak exists even when no $D_1$ peak is present$^{[9]}$ that is, without any kind of disorder as it involves ring breathing. The $D_1 + G$ peak is a combination of the $D_1$ and $G$ modes. There is also the first overtone of the $D_2$ and $D_4$
modes at 3,240 and 2,450 cm$^{-1}$ named $2D_2$ and $2D_4$, respectively. As a result of broadening Raman line width in the more disordered structures, their second-order Raman bands largely overlap each other. To this aspect, INR-A carbon black could be seen having the most ordered structure in our samples, whereas the OP6 soot has the least ordered structure.

It should be noticed that there is a discrepancy among published papers in the used nomenclature to define the Raman peaks. Its history could be seen in the supplementary of Ferrari and Basko$^{[25]}$ and in the review paper Merlen et al.$^{[26]}$ Throughout our Raman studies, we use $D_1$, $D_2$, $D_3$, $D_4$, and $G$ for the first-order peaks and $2D_4$, $2D_1$, $D_1 + G$, and $2D_2$ for the second-order peaks whose positions and origins have been mentioned above.

In Figure 1, also a Raman signal from the air in the Raman setup is shown. As the same probe volume of air is present in all our experiments, this signal will be additionally detected in all spectra in Figure 1. Therefore, these detected $O_2$ and $N_2$ peaks can be used as references for polarization effects and in comparing Raman intensities of various soot types. Notice that the detected signals are rotational-vibrational Raman signals of $O_2$ and $N_2$. They should contain O, S, and Q branches. Because of the weak signals, O and S branches could not be observed. We only see Q branch of $O_2$ and $N_2$ in our Raman spectra.

The differences in the Raman spectra of various LAC exhibit their structural dissimilarity. In order to understand more about the structural information, we decompose these spectra. The fitting protocol for the first-order Raman bands was discussed in Section 4.1.1 in the previous paper.$^{[6]}$ The second-order Raman bands is a combination of four Lorentzian curves assigned to the $2D_4$, $2D_1$, $D_1 + G$, and $2D_2$ peaks. In addition, $O_2$ and $N_2$ contribution is also fitted by Lorentzian function. Figure 2 plots the Raman spectral deconvolution for one carbon black (INR-A) and one flame soot at HAB = 8 mm after subtracting the fluorescence background. Besides the featured peaks of disordered carbonaceous materials such as $D_1$, $D_3$, $D_4$, $G$, and their second-order Raman peaks, the number and type of additional peaks reflect structural and chemical properties of LAC related to its formation process. In particular, the INR-A carbon black has a spectrum showing fewer peaks, and they appear generally narrower than the peaks in the flame soot sample. The intensity ratio between $D_1$ and $G$ peaks indicates a larger polyaromatic unit size for the INR-A sample in comparison with the flame soot sample.$^{[9]}$ For comparison, a decomposed OP6 spectrum is presented in Le et al.$^{[6]}$ showing a complex structure with contributions from oxygenated hydrocarbons, carbon chain structures, and organics.

The contribution of additional shoulder peaks leads to the increased width of the graphitic and disorder bands, expressed as full width at half maximum (FWHM). It should be noted that the “defect” $D_2$ peak is not separated from the $G$ peak in our analyses. With more defects and olefinic chains in the soot structure, the $G$ peak position gradually shifts towards higher Raman shift.$^{[27]}$ The presence of defects and disorders in our samples will also shorten the phonon lifetime and, correspondingly, broaden the linewidth. The degree of disorder of the samples could thus be estimated from the FWHM of the $D_1$ and $G$ peaks. The larger the FWHM is, the more disordered the structures are.$^{[27]}$ This is also true for the second-order bands of the Raman spectra because they are the combinations and overtones of the first order. Notice that for Raman spectra of graphite, the broadening of the second harmonic $2D_1$ (at $\sim 2,700$ cm$^{-1}$) is also associated with the stacking order occurring along the c axes.$^{[10,29]}$ This may also happen on our materials. With highly disordered structures, deconvolution of the component peaks in the second-order bands is difficult. Furthermore, the vibrational wavenumber of the $G$ peak is influenced by the purity of the material.$^{[29,30]}$ Therefore, this peak is also considered as a criterion for evaluation of the disorder.

### 3.2 | Polarization dependence

In Figure 3, we compare Raman spectra of each type of LAC in parallel and perpendicular polarization
Comparing the G-peak-intensity-normalized spectra, we see that the spectra from the parallel polarization (black curve) and the perpendicular polarization (green curve) too large degree overlap. A main difference can be observed at the $D_1$ peaks, where the more “ordered” LAC samples show a relatively low $D_1$ peak relative the $G$ peak for perpendicular configurations. More specifically, the heat-treated carbon black (INR-A) shows a relatively low $D_1$ peak in perpendicular configuration than a nonheat-treated carbon black (CXN-550), the more mature soot at the higher flame height shows a relatively low $D_1$ peak in perpendicular configuration than the young soot at lower flame height, and the more mature soot (OP1) produced from the mini-CAST soot generator has a relatively low $D_1$ peak in perpendicular configuration than the more immature soot (OP6). Another observation is that there are some polarization effects also on the second-order peaks $2D_1$ and $D_1 + G$ that will be further analyzed in relation to Figure 5.

The Raman peak from nitrogen is clearly observed for the parallel polarizations at 2,330 cm$^{-1}$ but with different intensity in the normalized spectra of Figure 3. The absolute N$_2$ signal is the same for all measurements and can be used for calibration of absolute intensity. Hence, the apparent absence of the N$_2$ peak for the OP6 mini-CAST sample is just because of the very strong Raman signal. The observed vibrational Raman signals for O$_2$ and N$_2$ are strongly polarized, and consequently, they are absent for the Raman setup with perpendicular polarization detection (red curve).

When taking the intensity ratio between the $D_1$ and $G$ peaks in the parallel polarization geometry ($I_{D_1}/I_G)_p$ and that in the perpendicular one ($I_{D_1}/I_G)_p$, we found different ratios for the LAC samples. By forming the ratio ($I_{D_1}/I_G)_p/ (I_{D_1}/I_G)_s$, and plotting that versus the $G$ peak position, an interesting correlation was found as shown in Figure 4a. It should be recalled that not only FWHM but also the $G$ peak position could also be disorder representatives.[27] The FWHM of $G$ band of graphene becomes narrower by three wavenumbers in perpendicular polarization compared with the parallel polarization.[15] Although this trend could not be seen in our samples due to the limited spectral resolution of 12 cm$^{-1}$, this should be noticed. We thus prefer to suggest the $G$ position as a better indicator of the disorder in the polarization study. As described previously, the $G$ peak position moves towards lower Raman shift for more ordered structures due to the domination of $E_{2g}$ symmetry mode of aromatic rings, less contributions from the “defect” $D_2$ peak at 1,620 cm$^{-1}$, and stretching mode of olefinic chains.[27] In addition, this band position is sensitive to clustering of the sp$^2$ phase, bond disorder, and the way sp$^2$ and sp$^3$ carbons coupled to aromatic carbons.[9]

Our result shows that ordered structures with lower

**Figure 3** Raman spectra of light-absorbing carbonaceous (LAC) obtained for parallel (black curves) and perpendicular (red curves) polarization geometries. Green curves are the normalization of red curves with black curves at $G$ peak
peak Raman shift have larger difference between \( I_{D1}/I_G \) for the different polarizations, and as shown in Figure 4a, it seems possible to establish a linear correlation between this ratio and the \( G \) peak Raman shift. This is an evidence

**FIGURE 4** The relationship of \( D_1 \) and \( G \) peak intensity ratio (a) and depolarization ratio \( D_p \) (b) as a function of the Raman shift of the \( G \) peak. (c) The depolarization ratio \( D_p \) as a function of full width at half maximum (FWHM) of \( G \) peak. HOPG, PG, GC, GP1, and GP2 are the highly oriented pyrolytic graphite, pyrolytic graphite, and glassy carbon, respectively, in Yoshikawa et al.’s study\(^{[16]}\) and monolayer and bilayer graphene in Sahoo et al.’s study\(^{[15]}\).

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**FIGURE 5** Polar plot for the peak intensity of peaks related to various vibrational modes in Raman spectra of light-absorbing carbonaceous (LAC) samples and air as a function of the relative angle of detected polarization and polarization of incident laser beam.
that the polarization effects in Raman spectra of disordered carbon-based materials need to be seriously studied because the intensity ratio of $D_1$ and $G$ peak is an important parameter often used in evaluating polyaromatic unit mean size of these materials.\cite{31,32} Hence, neglecting polarization effects could cause uncertainty in the evaluation of Raman spectra of carbonaceous materials.

The assignment of Raman lines to particular vibrational modes is added by noting the state of polarization of the scattered light. The depolarization ratio ($D_p$) can be expressed as the intensity ratios of the scattered light with polarizations perpendicular and parallel to the plane of polarization of the incident light, $D_p = I_d/I_p$, where $I$ is the peak intensity. For the $G$ peak, $D_p = 1$ for monolayer and bilayer graphene\cite{15} and also for highly oriented pyrolytic graphite.\cite{16} This value changes in suspended bilayer graphene\cite{15} and decreases with the decrease in the crystallite size of the graphic plane.\cite{16} A lower $D_p$ value corresponds to a higher Raman shift of the $G$ band implying no longer the $E_{2g}$ symmetry, suggesting that each crystallite is broken and converted into small sp$^2$ clusters.\cite{16} Therefore, the correlation of the $D_p$ and $G$ peak position is a feature of sp$^2$ carbon clusters in graphite materials. This trend is also observed in our study on LAC. As seen in Figure 4b,c, the observed $D_p$ of the $G$ mode decreases from 0.56 to 0.46 with the higher Raman shift of $G$ peak and larger width of $G$ peak corresponding to more disordered LAC materials. The depolarization ratios for the $G$ peak of our LAC materials varies from less disordered (INR-A carbon black) to much more disordered (OP6 sample) but still far from that of amorphous carbon a:C–H ($D_p = 0.37$).\cite{16}

Figure 5 exhibits the intensity variation of C–H, $D_1$, $G$, 2$D_2$, and $D_1 + G$ peaks in Raman spectra of LAC (Figure 5a–f) and the corresponding of N$_2$ and O$_2$ from the air (Figure 5g) versus the relative polarization between the incident light and the observed scattered light. In general, the peak intensity decreases when the polarization analyzer is rotated gradually from the parallel geometry to the perpendicular geometry. However, the profiles of the intensity variation are slightly varied from each type of sample.

A Raman line is classified as depolarized if it has $D_p$ close to or greater than 0.75 and as polarized if $D_p$ is smaller than 0.75.\cite{33} Only totally symmetrical vibrations give rise to polarized lines in which the incident polarization is largely preserved.\cite{33} Obviously, the depolarization of $D_1$, $G$, 2$D_1$, and $D_1 + G$ bands varies from 0.3 to lower than 0.75, whereas the depolarization of C–H “out-of-plane” mode ($\sim$850 cm$^{-1}$) is much lower. The depolarization of the C–H mode behaves similarly as the peaks of O$_2$ and N$_2$ molecules in the air (Figure 5g). This means that it emits Raman scattering in the same polarization orientation as the incident light. When the analyzer is aligned with the laser incident light, the maximum Raman scattering is observed. When the analyzer is crossed, the intensity of the linearly polarized Raman scattering is extinguished ($I_s = 0$). Its depolarization ratio thus approaches zero ($D_p = 0$).

The peak intensities for our analysis in Figures 4 and 5 were taken from the maximum value of each peak in the real spectrum after subtracting fluorescence, thus without spectral deconvolution. This is considered to be reasonable for polarization analysis of LAC in our study. In fact, the C–H peak at $\sim$ 850 cm$^{-1}$ is spectrally quite isolated and located far from other peaks in Raman spectra of LAC so its intensity will be unchanged whether the spectral deconvolution is made or not. However, with $D_1$ and $G$ peak intensities in Raman spectra of LAC, especially for the more disorder samples, the contribution and overlap of many peaks from various vibrational modes lead to high uncertainty in spectral deconvolution. A comparison was done for the two samples INR-A carbon black and ethylene/air soot at HAB = 8 mm resulting similar behaviors between real peak intensity and fitted peak intensity. The real peak intensity was finally selected for studying the polarization behavior of peak intensity of all various LAC materials.

4 | CONCLUSIONS

For Raman spectroscopy of LAC in backscattering configuration, the spectral information shows some differences due to the relative polarization of the incident light and the observed scattered light. For more ordered carbonaceous structures, these spectral differences increase. An important implication of this study is thus to emphasize that a clear statement of the polarization properties of a Raman setup is crucial for accurate characterization of carbonaceous materials and consequently to be able to compare results from Raman studies presented in scientific literature. Our polarization study also demonstrates that the major peaks in Raman spectra of LAC are due to totally symmetric vibrations as their depolarization ratios ($D_p$) are lower than 0.75. The O$_2$ and N$_2$ in the air of the Raman setup gave strongly polarized Raman signals that were used both for signal calibration and for certifying correct polarization properties of the Raman setup.

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CONFLICT OF INTEREST
The authors declare that they have no competing interests.

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