



To optical properties of carbon nanoparticles: A need in comprehending Urbach energy



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ABSTRACT

In this work, light absorption spectra of carbon nanoparticles sampled at different heights from an ethylene/air premixed flame were studied. The spectral behavior of the light absorption coefficient was analyzed in the high energy (Tauc) and low energy (Urbach) regions. In the Tauc region, information on the optical band gap can be retrieved, while in the Urbach region the value of the Urbach energy can be estimated. The variation of the spectral behavior of the light absorption coefficient in the low energy region was ascribed to different Urbach energies of the material depending on the sampling height. The Urbach energy is related to the disorder of the particles under analysis. The conclusion was made that due to the strong tail absorption, the traditional optical band gap concept is not an accurate approach in describing properties of studied nanocarbons, which can be better emphasized with the Urbach parameter. Moreover, the high value of the Urbach energy obtained is hypothesized to occur due to the fast nanoparticle formation in a flame that leads to a strong structural disorder. A thorough study of the Urbach energy and its relation to the nanoparticle growth looks an essential step in comprehending optical properties of carbonaceous particulate generated in flames.

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1. Introduction

Carbon nanoparticles resulting from an incomplete combustion processes are of wide interest for the combustion community for their impact on human health and environment. Understanding carbon particle formation in flames is essential in order to control the carbon pollutant emission. Recently, this combustion by-product is receiving particular attention to be employed and enhanced in different applications, thanks to the specific physico-chemical properties related to their structure and composition [1].

In this context, the optical properties of these carbon nanoparticles are widely investigated for different applications [2,3]. Optical properties have a significant impact on climate due to the material role in the radiative energy balance in the atmosphere [4]. Moreover, they are also required for an accurate diagnostics in the study of particles formation in sooting flames [5–9]. Recently, the major research is focused on the determination of the optical band gap of

carbon nanomaterials that is a parameter related to the different hybridization of the carbon atom wave function, sp^3 and sp^2 (being sp considered absent in these systems) [10–13]. Therefore, information on the maturity (C/H ratio) as well as degree of graphitization of these nanoparticles can be retrieved [14].

The optical band gap measurements are based on processing the light absorption spectrum in the UV–vis–NIR spectral range using the Tauc plot [15]. It is important here to underline that the value inferred from these measurements is not the absorption edge, which, however, can be obtained differently [16]. Concerning the nanoparticles under analysis, the application of Tauc plot processing is well-known to have inherent drawbacks. Specifically, 1) the type of electron transitions responsible for light absorption should be known *a priori* since it determines the exponent entering the Tauc law, as reported in the next section; 2) the choice of the linear region of the plot is somehow subjective - there is no a clear way to distinguish between a tangent to the corresponding curve at an arbitrary point and the actual linear dependence. Another issue, which has not been widely discussed, is related to the nature of light absorption of carbon particles at flame temperatures. Besides the fundamental absorption being the basis of the Tauc procedure, light absorption can occur on free electrons that

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appear due to thermal excitation. In the latter case, results of the Tauc processing might be questioned even if the inherent issues are addressed.

In the current paper, we focus on the other optical parameter of carbon nanoparticles, namely, the Urbach energy. This parameter being an essential part of the Tauc formalism, has not been paid enough attention. At the same time, the Urbach energy rather than the optical band gap can determine the magnitude of light absorption of the material in the visible and infrared regions. In order to reduce a potential contribution of thermally excited electrons, we characterize the cold airborne particulate after sampling it from a sooting flame. A comparison of the spectral behavior of the measured light absorption of nanoparticles sampled at different heights allowed for conclusions on the Urbach energy variation vs. the height (i.e., the stage of formation). Based on the paper findings, further research on understanding the mechanism of the Urbach energy development during nanoparticle formation looks an essential step in an accurate modeling of optical properties, and therefore, the radiative transfer in the carbon nanoparticles generated in flames.

2. Light absorption analysis

Light absorption analysis can be performed based on the energy level scheme described by Tauc and Menth [15]. Two characteristic regions can be distinguished in the absorption spectrum. The high energy region that originates from the electron excitations from the valence to the conduction band is called the Tauc region, while the low energy absorption in the Urbach region arises from tail states in the forbidden band. The spectral dependence of light absorption coefficient, α , on the energy of an absorbed photon, E , can be expressed in the Tauc region as

$$\alpha_T(E) \propto \frac{(E - E_T)^r}{E}, \quad (1)$$

and in the Urbach region as

$$\alpha_U(E) \propto \exp\left(\frac{E}{E_U}\right), \quad (2)$$

where the subscripts T and U denote the corresponding regions. E_T is the Tauc energy, i.e., what is coined the optical band gap, and E_U is the Urbach energy. The parameter r is a constant that depends on the type of inter-band electron transitions. Note that since we are interested in the spectral behavior of light absorption coefficient, we can omit all energy-independent variables in Eq. (1), (2). It should be added that the expression of the light absorption coefficient in the form Eq. (1) is well-known and widely used. Eq. (2) is a consequence of the Urbach rule [17]

$$\alpha(h\nu) = \alpha_0 \exp\left[\frac{h\nu - E_g(T)}{E_U(X, T)}\right], \quad (3)$$

where α_0 is the constant, $E_U(X, T)$ is the Urbach energy that depends on static (X) and dynamic (T) disorder degrees of the material and $E_g(T)$ is the energy gap as function of temperature. Then, Eq. (3) directly transforms into Eq. (2) with the photon energy, $E \equiv h\nu$.

The traditional approach to the light absorption processing is fitting the measured data to Eq. (1) in order to determine the Tauc energy. This fitting requires assumptions on the value of the power r , although the recent study claims the possibility to infer both E_T and r [18]. The major issue with this approach is that the validity of Eq. (1) within the entire fitting region is essential due to a high sensitivity of the procedure. As a result, the accuracy in finding the energy band gap becomes related to the stability of the fitting procedure. At a given r , the fitting method could be reduced to find a linear region in the plot of $[\alpha(E)E]^{1/r}$ vs. E . The Tauc energy is the

intercept of the corresponding line with the abscissa axis [12]. In order to distinguish between an actual linear region and a tangent to the plot at a certain energy one should ensure that at low energies the plot curves up compared to an extrapolating line. Also, an expected variation in the energy gap due to a change in particle properties should appear as a result of a parallel shift of the extrapolating line [19] and not to originate from different line slopes. These two essential criteria of the Tauc procedure validity are not always considered while studying carbon particles that results in some misinterpretation.

This traditional approach of data processing makes sense in the case of a well-defined forbidden energy band. On the contrary, at the occurrence of long energy tails, i.e., at an existence of a substantial number of energy states within that forbidden band, the energy gap concept loses its meaning as an energy below which electron transitions do not occur. Then, a logical approach to consider for absorption analysis is to estimate the Urbach energy, and correspondingly, to conclude on the role of the tail light absorption before applying the Tauc plot. Applying the Urbach plot, i.e., reporting the measured absorption in a semi-log scale as $\ln(\alpha)$ vs. E , one can obtain the slope of the curve at low energies [17,20,21]. This slope is a reciprocal of the Urbach energy, which can be then easily evaluated. If the Urbach energy is high enough, i.e., the sharp energy gap smears, a study of the Urbach energy itself rather than attempts to infer that remnant energy gap is the best approach to get insight into material optical properties.

3. Experimental

The experimental set-up employed for absorption measurements is shown in Fig. 1. Measurements were carried out on carbon nanoparticles produced and sampled from a rich premixed ethylene/air flame. A bronze McKenna Burner is used, with the stabilizing stainless-steel 60 mm diameter plate positioned at 30 mm height above the burner. The flame under analysis is characterized by $C/O = 0.67$, an equivalence ration of $\varphi = 2$, and the gas velocity of 10 cm/s.

The particles are sampled from the center of the flame through a 2 mm diameter hole of a horizontal stainless-steel sampling probe (10 mm O.D. and 8 mm I.D.) into the measuring cell. A controlled quantity of Nitrogen is flowed in the sampling probe and particles aerosol is collected with a dilution of about 10, in order to quench chemical reactions and dilute the aerosol still assuring a good sensitivity in absorption measurements.

The absorption-measuring unit is similar to the one described in our previous work [13] and here briefly recalls for clarity. The measuring cell consists of a 450 cm cylindrical polycarbonate tube,

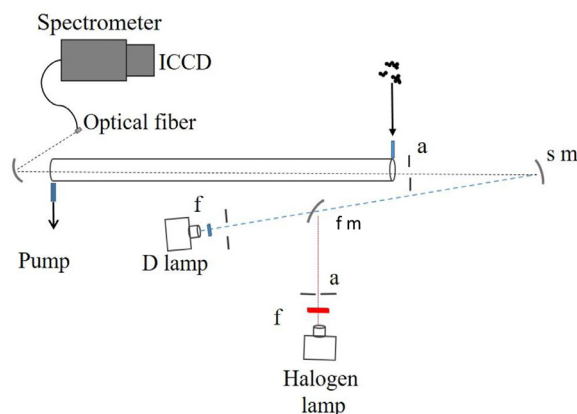


Fig. 1. Experimental set-up for absorption measurements (f m: flip mirror, f: filter, a: aperture, s m: Schlieren mirror).

longer than the tube used in [13] in order to increase the detection limit. The tube is closed at its ends with two quartz windows (50 mm diameter). A Schlieren mirror (1534 mm focal length, s m in Fig. 1) allows sending the collimated light beam across the tube. The light is then focused on an optical fiber (Oriel, 3 mm bundle diameter) by means of a second spherical mirror (120 mm focal length). To perform UV-vis - near IR extinction measurements the following sources and detectors are employed to cover the wavelength range of interest.

- In the 280 nm - 530 nm spectral range a Deuterium lamp (63,379 Oriel 30 W) is used as light source. Signal intensity is measured with a Czerny-Turner spectrograph (Shamrock 303i, 150 grooves/mm) coupled with an ICCD camera (iStar 334T, Andor Technology).
- At longer wavelengths (530-850 nm) we change the light source with an intense diffuse Halogen lamp (400 W) and we use the same spectrometer for signal detection.

A flip mirror (f m in Fig. 1) is used in order to select alternately Deuterium and Halogen lamp. To increase signal to noise ratio, each spectrum results from an average over 500 samples. In order to eliminate possible contribution of gas species to total absorption, a glass fiber filter is positioned before the measuring unit to stop the particles flow in the tube. The resulting spectrum was subtracted to the particle absorption spectrum. The on-line approach proposed allows performing a fast analysis of the nanoparticles before collection for possible post-treatment tool.

It should be emphasized that our experimental approach, which allows for measurements of light absorption of cold particles, is preferential compared to conventional measurements of hot particles in flames provided that the peculiarities of fundamental light absorption are in focus.

4. Results and discussion

According to the Beer-Lambert law, the spectral transmittance τ_λ , which is defined as the ratio of transmitted $I_{\lambda,T}$ and incident $I_{\lambda,0}$ light intensity at a given wavelength, λ , can be expressed as

$$\tau_\lambda \equiv \frac{I_{\lambda,T}}{I_{\lambda,0}} = \exp\left(-\int_0^L K_{ext,\lambda} dx\right) \quad (4)$$

where $K_{ext,\lambda}$ is the extinction coefficient of the particles in the aerosol, x the spatial location along the extinction pathway, and L the optical pathlength. The extinction coefficient is due to the sum of absorption and scattering coefficient. Considering the scattering contribution negligible [9], Eq. (4) can be re-written in terms of the absorption coefficient $K_{abs,\lambda}$ as

$$\tau_\lambda = \exp\left(-\int_0^L K_{abs,\lambda} dx\right) \equiv \exp(-Q_{abs,\lambda}L) \quad (5)$$

with $Q_{abs,\lambda}$ related to the particle volume fraction, f_V

$$Q_{abs,\lambda} \propto q(\lambda)f_V, \quad (6)$$

where the parameter $q(\lambda)$ is proportional to the light absorption coefficient of the material, $\alpha(\lambda)$.

Then, the parameter $q(\lambda)$ is proportional to the logarithm of the experimentally measured transmittance. Thus, the volume fraction and pathlength, which are not evaluated in this work, do not affect the spectral behavior of $q(\lambda)$, which is related to the measured transmittance as

$$q(\lambda) \propto -\ln(\tau_\lambda). \quad (7)$$

Then, based on Eq. (7), the parameter $q(\lambda)$ was obtained as the logarithm of the measured transmittance. For an easier comparison of the spectral behavior of this parameter measured in airborne

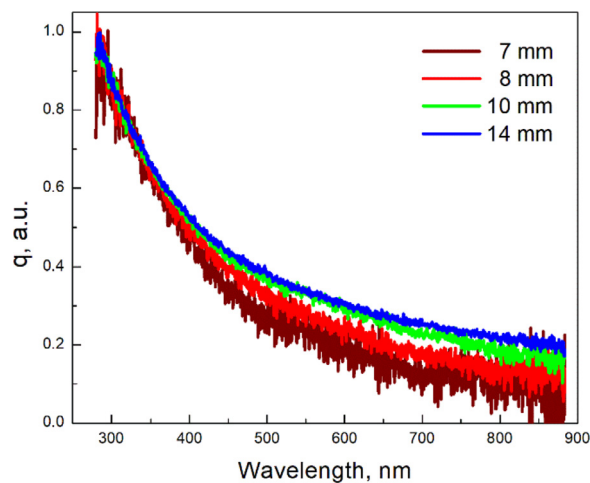


Fig. 2. Spectral behavior of the parameter q (that is proportional to the light absorption coefficient) in carbon nanoparticles sampled at different heights. .

carbon nanoparticles sampled at different heights, we normalized the short-wavelength part of spectra as plotted in Fig. 2. Then, the spectral difference is clearly seen in the long-wavelength region.

Due to their proportionality, i.e., $q(\lambda) \propto \alpha(\lambda)$, the actual light absorption coefficient of carbon nanoparticles has the same spectral behavior as that shown in Fig. 2 for the parameter $q(\lambda)$. Furthermore, the Urbach and Tauc plots of the parameter $q(\lambda)$, which are analyzed below, have the same meaning as they were processed for the actual light absorption coefficient $\alpha(\lambda)$ following Eq. (1), (2).

The spectral difference of the parameter $q(\lambda)$ in different nanocarbon samples is better distinguished in the Urbach plot shown in Fig. 3. A linear behavior of $\ln(q)$ vs. E is evident for all samples at energies between about 3.5 eV and 2 eV as seen in Fig. 4. Note that at energies below about 2 eV the noise-to-signal ratio becomes too high not allowing for definite conclusions.

The linear fitting performed in Fig. 4 allows estimating the Urbach energy of carbon nanoparticles collected at different heights. The Urbach energy calculated as a reciprocal of the line slope is reported in Fig. 5.

The obtained values of the Urbach energy suggest that in the case of data processing using the Tauc plot, the energy region

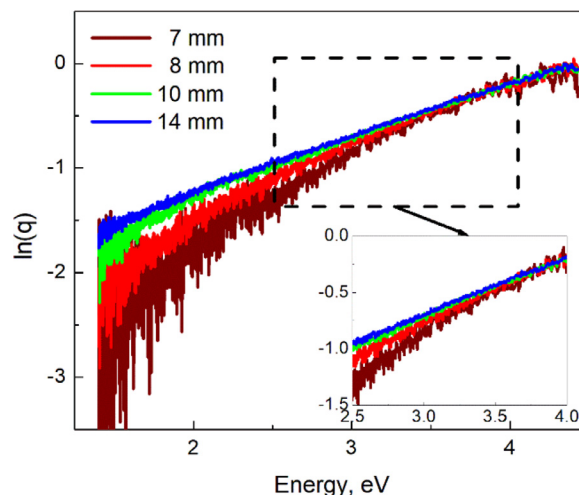


Fig. 3. Urbach plot of the parameter $q(\lambda)$ in carbon nanoparticles sampled at different heights. Inset shows the region, where the spectral difference begins to be evident.

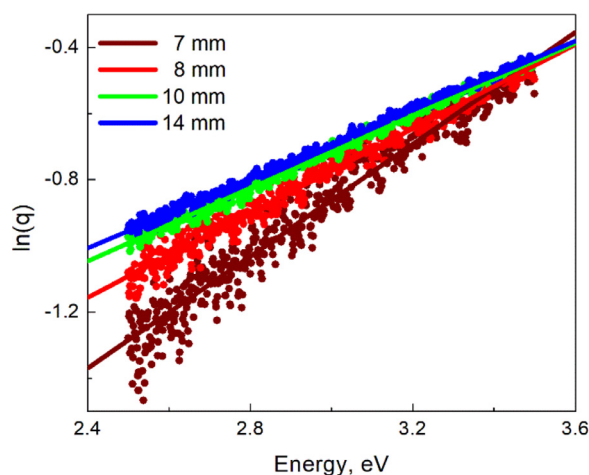


Fig. 4. Linear fitting of $\ln(q)$ vs. energy in the range 2.5 – 3.5 eV. Different curves refer to data of carbon nanoparticles collected at different heights. .

around 1.5 eV cannot be considered and the extrapolating line must be drawn from noticeably higher energies. In Fig. 6 we show two cases of this Tauc extrapolation, i.e., from low and high energies. The data processing is performed under assumptions on indirect transitions, i.e., $r = 2$ is chosen in Eq. (1), and, correspondingly, $[q(E)E]^{1/2}$ is plotted against energy.

In Fig. 6, the intercepts of extrapolating lines with the abscissa axis give the following values: 1) about 0.95 eV for the 7 mm sample and about 0.07 eV for the 14 mm sample if lines are drawn from low energies; 2) about 1.4 eV for all samples if lines are drawn from high energies. As we already mentioned, provided high values of the Urbach energy, i.e., a long-tail light absorption measured in carbon nanoparticles, the energy values obtained using the Tauc plot in Fig. 6 do not correspond to the optical band gap concept. The gap is an energy below which the light absorption significantly drops, which is not what occurs in our case. Then, probably the gap value is a less useful parameter in describing optical properties of nanocarbons than considered up to now.

It worth noting that even if the energy gap value appears to be not that appropriate, its inaccurate processing may lead to some misinterpretations. As shown in Fig. 6, if the extrapolating line is drawn from low energies, the different intercepts are due to differ-

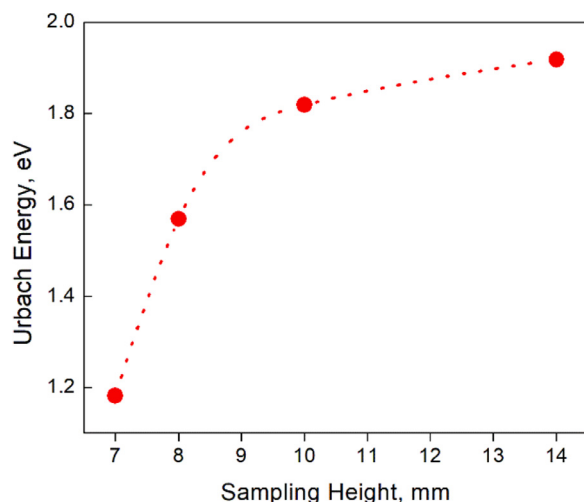


Fig. 5. Urbach energy of carbon nanoparticles sampled at different heights. The dotted line is a guide to the eye. .

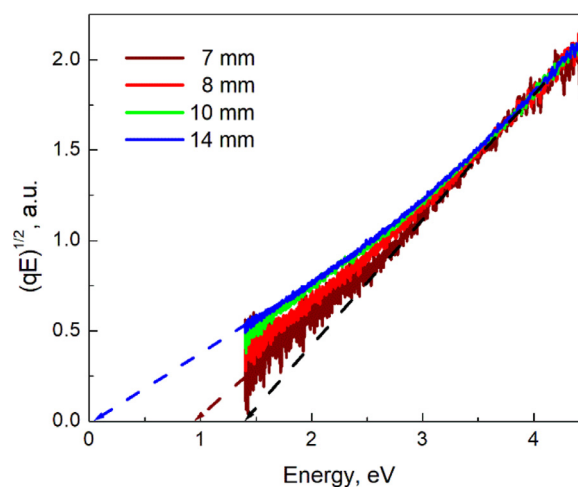


Fig. 6. Tauc plot for the parameter $q(\lambda)$ in carbon nanoparticles sampled at different heights. Indirect transitions are assumed. The linear extrapolation from low energies is different depending on the sampling height. The linear extrapolation from high energies is indistinguishable for different samples.

ent slopes of the Urbach tails. In this case, the obtained values that are supposed to be the optical band gaps are meaningless.

The origin of high Urbach energies reported in Fig. 5 requires a detailed study. Usually, the Urbach tails are ascribed to the material disorder. We suggest that this structural disorder can be developing during nanoparticle formation. In our previous work, considering nano-oxides generated in metal-containing flames, we discussed that the disorder is related to a local charge imbalance due to oxygen deficiencies [22,23]. This disorder that leads to the Urbach energy on the order of 1.5 eV is the reason of their strong light absorption in the visible range, so the wide-gap dielectric nanoparticles become strong light emitters while being generated in flames.

We hypothesize that the high Urbach energy in nanocarbons could be of similar nature. The substantial disorder originates from the fast nanoparticle growth in flames. In our opinion, at least two kinds of disorder are possible in nanocarbons, namely 1) an occurrence of broken bonds, i.e. local defects such as carbon atom deficiencies that, for example, may exhibit themselves as non-closed rings in the case of sp^2 hybridization, and 2) a relative slippage of stacked graphene layers within a nanoparticle. More work is needed to get insight into these possible types of disorders.

5. Concluding remarks

In this work, we measured light absorption of carbon nanoparticles sampled from a flame at different heights. The performed analysis of the spectral behavior of the light absorption coefficient demonstrates the importance of using Urbach energy to describe optical properties of nanocarbons. The high Urbach energies obtained in this study can invalidate the optical band gap concept in describing light absorption in the material of interest. Thus, our results justify a need in emphasizing the Urbach energy more than the optical band gap energy being the subject of research for a long time.

It is important then to understand the reason of such high values of the Urbach energy and its relation with other material properties, which should be addressed in a future work. A detailed study of light absorption in carbon nanoparticles focused on the Urbach energy combined with the thoroughful characterization of the material disorder is an essential step to get insight into the problem. A fresh look at the theoretical description of light absorption in disordered carbon materials is also required.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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