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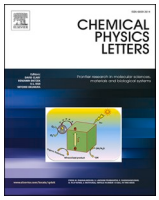

Photoexcited state properties of *N*-ethylcarbazole-functionalized carbon dots in solution and in PVK polymer matrix

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A B S T R A C T

The adducts of *N*-ethylcarbazole (NEC) to small carbon nanoparticles prepared by microwave-assisted thermal processing are carbon dots (CDots) with NEC for the dot surface functionalization, thus NEC-CDots, which were shown previously for their high stability in the dot structure and properties. In this study, the optical absorptions and photoexcited state properties of NEC-CDots in solution and in poly(*N*-vinylcarbazole) (PVK) film matrix were evaluated by using optical spectroscopy methods, including those for fluorescence decays and lifetimes. The results suggest that the properties are largely unchanged from solution phase to the polymer matrix environment, very positive to their expected optoelectronic applications.

1. Introduction

Carbon dots (CDots) [1–3], which are defined as small carbon nanoparticles (CNPs) with effective surface passivation [1–3], have captured much recent attention as evidenced by the rapidly increasing number of relevant publications [4–15]. The relationship between CNPs and CDots in terms of their optical spectroscopic properties is such that they are similarly colored with significant optical absorptions in the visible spectrum. For CDots in which the core CNPs are surface functionalized with colorless organic molecules, their optical absorption spectrum is generally very similar to that of the CNPs [16], indicative of the insensitive nature of their photon harvesting characteristics. However, upon photoexcitation, the excited state properties of CNPs and CDots are majorly different, as reflected by their very different fluorescence emission behaviors. CNPs in solvent dispersions are only weakly emissive, with observed fluorescence quantum yields generally less or much less than 0.5 % [17,18]. CDots on the other hand display bright and colorful fluorescence emissions with quantum yields easily above 10 %, up to 70 % in some dot configurations [3,19]. The dramatic enhancement in fluorescence emissions is attributed to the organic functionalization in CDots that effectively passivates the surface of CNPs to prevent or reduce the “leaking” of photoexcitation energy through the surface defects of CNPs, which is analogous, both in principle and in actual outcome, to the surface capping of classical semiconductor nanoparticles by another wider bandgap semiconductor for core/shell nanostructures such as the famous CdSe/ZnS quantum dots (QDs) [20–23].

Among the many organic molecules used for the functionalization of CNPs, *N*-ethylcarbazole (NEC) is of special interest in several respects. Under thermal processing conditions including especially those associated with the microwave irradiation that is preferentially absorbed by CNPs, NEC radicals can be generated for their *in situ* addition to the CNPs for NEC-CDots (Fig. 1) of high stability in structure and properties [24,25]. NEC molecule is structurally almost identical to the individual units in poly(*N*-vinylcarbazole) (PVK), which is an important electronic polymer of broad technological applications, including especially those in electronic and optoelectronic devices [26–29]. The excellent compatibility of NEC-CDots with PVK is particularly valuable to the fabrication of high-quality PVK/NEC-CDots composites. The uses of such composites in nano-optoelectronics should benefit from the efficient photon harvesting and bright fluorescence emissions of CDots and also their unique photoexcited state redox characteristics [24].

The studies of CDots-derived polymer nanocomposites are scarce in general, even more so for those based on well defined and characterized CDots from the deliberate chemical functionalization synthesis and with optoelectronically valuable polymers. In this work, the photoexcited

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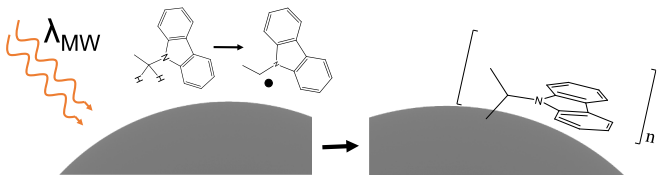

Fig. 1. Cartoon illustration on the microwave-assisted generation of NEC radicals on the carbon nanoparticle surface for *in situ* addition to form NEC-CDots.

state properties of NEC-CDots in solution and in PVK film matrix were evaluated by using optical absorption and fluorescence spectroscopy methods, including fluorescence decays and lifetimes. The results are analyzed and compared, and their mechanistic implications are discussed.

2. Experimental section

2.1. Materials

The carbon nanopowder sample was supplied by US Research Nanomaterials, Inc. *N*-ethylcarbazole (99 %) and 1,2-Dichlorobenzene (>98 %) was purchased from Beantown Chemical, poly(*N*-vinylcarbazole) (molecular weight ~ 90,000), THF, and DMF (>99.7 %) from Acros Organics, and toluene from Burdick & Jackson.

2.2. Measurement

UV/vis absorption spectra were recorded on a Shimadzu UV2501-PC spectrophotometer. Fluorescence spectra were acquired on a Jobin-Yvon emission spectrometer equipped with a 450 W xenon source, Gemini-180 excitation and Triax-550 emission monochromators, and a photon counting detector (Hamamatsu R928P PMT at 950 V). Fluorescence decays were collected via time-correlated single photon counting (TCSPC) technique on a Horibia Ultima Extreme spectrometer equipped with a SuperK Extreme supercontinuum laser source pulsed at 5 MHz, TDM-800 excitation and TDM-1200 emission monochromators, a R3809-50 MCP-PMT detector operated at 3.0 kV in a thermoelectrically cooled housing, and FluoroHub A + timing electronics. The time resolution of the measurements, as characterized by the instrumental response function (IRF) of the setup, is 100–200 ps (depending on excitation wavelength). Experimental decay curves were fitted with Das6 fluorescence decay analysis software.

2.3. Nec-cdots and dispersion in PVK films

The synthesis and characterization of NEC-CDots have been reported previously [24,25], and the relevant details are provided in the Supplementary Materials.

The optically transparent PVK films with dispersed NEC-CDots were prepared by mixing NEC-CDots and PVK in solution for wet-casting. Solutions of PVK (molecular weight ~ 90,000) in dichlorobenzene and NEC-CDots in DMF were prepared. The two solutions were mixed, and the resulting mixture was degassed, concentrated via the evaporation of the solvents, and then drop-cast onto a clean glass slide. Upon slow solvent evaporation, a visually homogenous composite film of NEC-CDots dispersed in PVK was obtained and used for spectroscopic measurements. It should be emphasized that the NEC-CDots were pre-made and fully characterized [24,25] before their dispersion into the PVK film matrix, not synthesized in the presence of any PVK polymer. The amount of NEC-CDots dispersed in the PVK film was to have the optical absorbance of ~ 0.1 at 400 nm for photoexcitation, thus very dilute, much less than 1 % by weight and even lower in terms of the number concentration of the dots, so that there is no possibility for any association or interactions between the dispersed NEC-CDots in the PVK polymer film matrix.

3. Results and discussion

NEC-CDots are adducts of NEC molecules to CNPs, with the likely involvement of NEC radicals due to the loss of an α-hydrogen, N-C**H**$_2$-CH$_3$ (Fig. 1), enabled by the microwave irradiation-assisted thermal processing conditions. The mechanistic details on the generation of NEC radicals on the surface of a CNP for *in situ* addition remain to be further explored and determined. Nevertheless, one may consider possibilities such that the "hot" surface of a CNP (due to its preferential heating by the microwave irradiation) could facilitate an NEC molecule in the close vicinity of the CNP losing an α-hydrogen, or that the microwave heating of a CNP may result in the generation of carbon radicals on the CNP surface, with some of the carbon radicals succeeding in the α-hydrogen abstraction from nearby NEC molecules to generate NEC radicals for additions to the CNP. Since the CNP surface is expected to be populated by abundant defects, one may even argue that the second possibility seems more reasonable. Regardless of the mechanistic details on the adduct formation, NEC-CDots are structurally stable and also stable in their optical spectroscopic and other properties against harsh treatments such as refluxing solutions of NEC-CDots in various solvents for an extended period of time [25]. The high stability of NEC-CDots in structure and properties is consistent with the likely NEC radical addition to CNP to form carbon–carbon bonds, which represents a unique functionalization scheme for the required effective surface passivation of CNPs in accordance with the classical definition of CDots.

NEC-CDots are soluble in various organic solvents, enabling solution-phase sample characterizations, such as the more quantitative ^{1}H and ^{13}C NMR results on the dot surface functionalization and structures [24,25]. On their solution-phase optical spectroscopic behaviors, shown in Fig. 2 is an optical absorption spectrum of NEC-CDots in solution. The spectrum is apparently similar to that of solvent dispersed CNPs, suggesting that the NEC functionalization of CNPs causes no significant changes to the CNPs' electronic transitions for the harvesting of visible photons. The molecular structure of NEC is nearly the same as the repeating unit in poly(*N*-vinylcarbazole) (PVK), a popular electronic

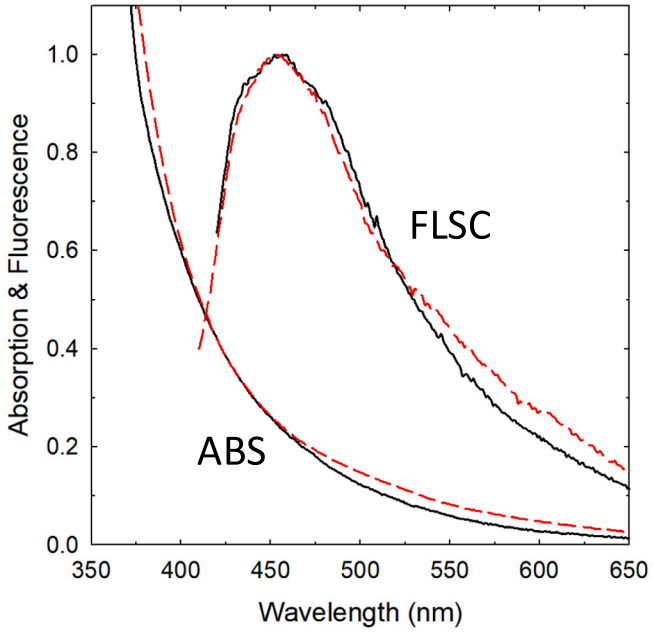

Fig. 2. Absorption (ABS) and fluorescence (FLSC, 400 nm excitation) spectra of NEC-CDots in THF solution (dashed line) and in PVK film matrix (solid line). The fluorescence spectra are corrected for nonlinear instrumental response with separately determined correction factors. The NEC molecule is colorless and has no optical absorptions in the near-UV to at least 350 nm, so it can not be excited in the spectral region for any fluorescence emissions.

polymer widely used in optoelectronics [26–29], and therefore NEC-CDots are fully compatible with PVK to allow the homogeneous dispersion of NEC-CDots in PVK polymer matrix. The resulting PVK/NEC-CDots composite films are optically transparent but colored due to the embedded NEC-CDots, comparable in film quality with the optically transparent and colorless neat PVK films. The optical absorption spectrum of NEC-CDots in PVK film matrix is generally similar to that in solution (Fig. 2), except for somewhat more light scattering effect in the film measurement. It seems safe to conclude that the electronic transitions in CNPs and CDots for the photon-harvesting in the visible spectrum are insensitive to different environments from solution-phase to the polymer matrix.

NEC-CDots in solution exhibit bright and colorful fluorescence emissions (Fig. 2). On the brightness, used in this study was a sample solution of 20 % in fluorescence quantum yield. For PVK/NEC-CDots composite films, it is generally rather difficult to get accurate fluorescence quantum yield results by using the relative method in reference to a fluorescence standard [30]. Nevertheless, intuitively there was no evidence for any major weakening of fluorescence emissions upon the dispersion of NEC-CDots into PVK film matrix. Such an assessment is also consistent with the fluorescence decay results of NEC-CDots in solution *versus* in composite films with PVK to be presented and discussed later in this manuscript.

Fluorescence emissions of NEC-CDots in solution are excitation wavelength dependent (Fig. 3), following the characteristic pattern that has been found for other CDots of different surface functionalizations [31]. For NEC-CDots in PVK film matrix, the same characteristic excitation wavelength dependence of fluorescence emissions was found (Fig. 3), suggesting that the photoexcited state properties of the CDots, at least those dictating emission color distributions, are not so sensitive to the environment of solution phase *versus* the polymer matrix. Thus, the same mechanistic rationale developed for the characteristic excitation wavelength dependence of fluorescence emissions for CDots in solutions [31] should also be applicable to that in the solid-state composite film environment.

The characteristic excitation wavelength dependence of fluorescence emission spectra is accompanied by the equally characteristic dependence of emission intensities such that the intensities or quantum yields decrease progressively with longer excitation wavelengths [25,31]. However, decoupled from such dependencies are fluorescence decays with excitations at different visible wavelengths, as shown in Fig. 4 for NEC-CDots in DMF solution. The observed decays are apparently much less sensitive to excitation wavelengths, generally with the longer excitation wavelengths corresponding to only slightly slower decays. All of the decays are non-exponential yet not too far from linear on the log scale (as illustrated in Fig. 4), and they could all be deconvoluted empirically with the use of a bi-exponential function, $I_F(t) = A_1\exp(-t/\tau_1) + A_2\exp(-t/\tau_2)$. Here the characterization of the decay curve deconvolution as "empirically" is for the likelihood that the use of the bi-exponential decay function represents an averaging of more than two decay components. Nevertheless, the fittings were generally successful, yielding two fluorescence lifetimes (τ_{F1} and τ_{F2}) and correspondingly two pre-exponential factors (A_1 and A_2) for each decay curve. In a further averaging, the pair of τ_F values and the corresponding A values were used to calculate the average lifetime $< \tau_F >$ for the observed fluorescence decay at a selected excitation wavelength, $<\tau_F> = (A_1\tau_{F1}^2 + A_2\tau_{F2}^2)/(A_1\tau_{F1} + A_2\tau_{F2})$ [30]. The $< \tau_F >$ values thus obtained are in the range of 5.6 ns to 6.6 ns for the different visible excitation wavelengths, not so significant variations.

For NEC-CDots in PVK film matrix, the excitation wavelength dependence of fluorescence decays is also not so significant (Fig. 5). The decay curves could similarly be deconvoluted by using the same bi-exponential function, which again represents the averaging of likely more than two decay components, followed by the further averaging to yield the average lifetime $< \tau_F >$ for each excitation wavelength. The $< \tau_F >$ values for NEC-CDots in PVK film matrix are apparently somewhat shorter than those in solution, in the range of 4.4 ns to 5.4 ns for the different visible excitation wavelengths, not so significant variations either. The somewhat smaller $< \tau_F >$ values of NEC-CDots in PVK film

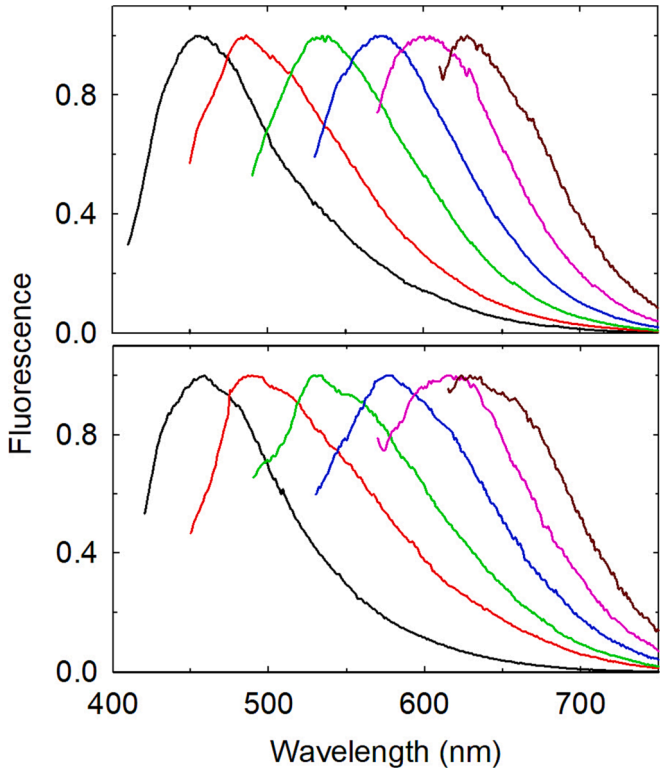

Fig. 3. Fluorescence spectra of NEC-CDots excited at 400 nm to 600 nm in 40 nm increments (from left to right) in THF solution (top) and in PVK film matrix (bottom).

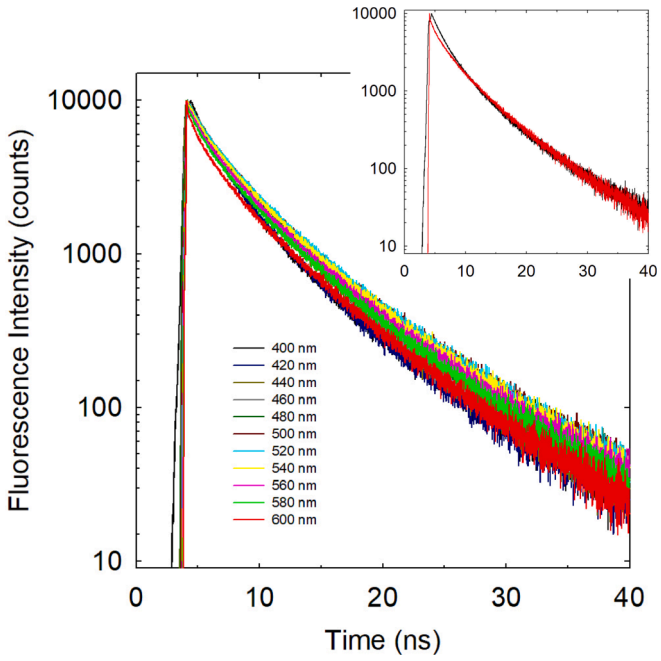

Fig. 4. Fluorescence decays of NEC-CDots in DMF solution with excitations at 400 nm to 600 nm in 20 nm increments. Insert: The decays at 400 nm (black) and 600 nm (red) excitations compared. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

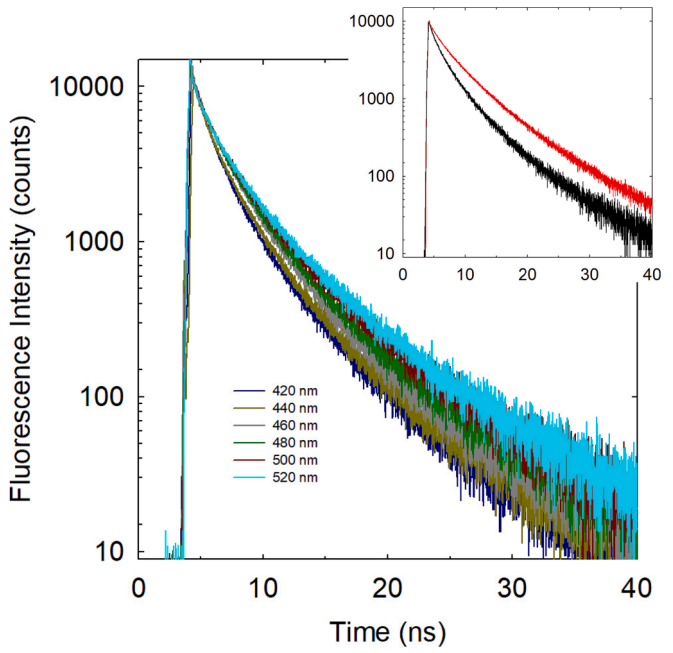

Fig. 5. Fluorescence decays of NEC-CDots in PVK film matrix with excitations at 420 nm to 520 nm in 20 nm increments. Insert: The decays in DMF solution (red) and in PVK film matrix (black), both at 500 nm excitation, compared. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

matrix than those in DMF solution might be attributed in the most part to the effect of medium refractive index (n) changes on the radiative rate constants (k_F) of the emissive excited states. According to the established photophysical principle there is ideally an n^2 dependence of k_F, namely $k_{F,film}/k_{F,solution} = (n_{film}/n_{solution})$ [2,30] thus $k_{F,film} > k_{F,solution}$ (and then $< \tau_F >_{film} < < \tau_F >_{solution}$) due to $n_{film} > n_{solution}$. Experimentally, however, the effect due to the change in medium reflective index has often been found to deviate from the more extreme n^2 dependence. Nevertheless, the effect is real and well established in the literature [30].

The rather similar fluorescence decays of NEC-CDots from the solution phase to the PVK polymer matrix suggest the absence of any meaningful energy and/or electron transfers between the CDots and PVK polymers, and also no association or interactions between the dispersed NEC-CDots in the PVK nanocomposite film.

PVK has been well established as a polymer for major applications in electronics and optoelectronics, such as the uses for hole transport and as photoconductor in various devices [26–29,32–34]. CNPs representing the nanoscale carbon allotrope at zero-dimension exhibit some of the same optical properties characteristic of other photonic nanomaterials including conventional semiconductor nanoparticles. The effective surface passivation mostly via chemical functionalization of CNPs for CDots is conceptually, phenomenologically, and to a significant extent mechanistically analogous to the surface capping of semiconductor nanoparticles by another nanoscale semiconductor for core/shell nanostructures or quantum dots (QDs), for which CdSe/ZnS is an excellent example. Thus, CDots for their optical properties and photoexcited state characteristics are often considered as being QDs-like, which has driven the growing interest in the development of CDots-based composites with electronic polymers [32–35], including the use of PVK polymers to functionalize CNPs for PVK-CDots [35]. A major advantage with PVK-CDots is the elimination of any foreign substance from the resulting PVK/CDots composites [36,37], though there are still issues and challenges such as the control of functionalization in the preparation of PVK-CDots, solubility limitations of the dot sample, and more difficult dot sample characterizations. Here the foreign substance refers to the molecules/species that are needed for the organic functionalization in CDots but may structurally significantly different from PVK, which could negatively impact the quality of the resulting nanocomposites due to possible issues of incompatibility between these molecules/species and the PVK matrix [36,37]. However, since NEC is nearly identical to the repeating unit in PVK polymers, thus without any significant compatibility issues, NEC-CDots serve as an excellent alternative to PVK-CDots for the desired nanocomposites of potentially significant applications in electronic/optoelectronic devices.

4. Conclusions

The results from this study show that the optical properties and photoexcited state characteristics of NEC-CDots in solution are typical of those found in other CDots of different surface functionalization schemes, namely no surprises at all. Equally interesting and significant is that the absorption and emission properties of NEC-CDots are largely unchanged from solution phase to the environment in PVK polymer matrix, thus justifying the value of the dot sample in potential electronic/optoelectronic applications. Also among major advantages of NEC-CDots are the more defined dot structure and the high dot structural stability (and by extension stabilities in optical and photoexcited state properties). Furthermore, because of the observed similarities between the observed properties in solution phase and polymer film matrix, the mechanistic framework developed and validated on the basis of CDots in solutions should be applicable to NEC-CDots and probably other CDots in solid state environments, which is important and valuable to the eventual technological applications of CDots.

CRediT authorship contribution statement

Weixiong Liang: Data curation. **Li Cao:** Data curation. **Annalise Scorzari:** Data curation. **Hannah McGrath:** Data curation. **Christopher E. Bunker:** Conceptualization, Supervision. **Xianyan Ren:** Data curation. **Ping Wang:** Methodology. **Liju Yang:** Conceptualization, Supervision. **Ya-Ping Sun:** Conceptualization, Supervision.

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.

Data availability

Data will be made available on request.

Acknowledgment

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.cplett.2023.140964.

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