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Photoluminescent Semiconductor Nanocrystals for Fingerprint Detection

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ABSTRACT: The concept of utilizing photoluminescent semiconductor nanocrystals for latent fingerprint detection, especially in concert with phase-resolved imaging for background fluorescence suppression, is reduced to practice with CdS nanocrystals that are capped with dioctyl sulfosuccinate. The nanocrystals are dissolved in heptane or hexane and are applied in much the same way as staining with fluorescent dye, on articles that have been pre-fumed with cyanoacrylate ester and also on the sticky side of electrical tape without pre-fuming. Since CdS can form a photoluminescent nanocomposite with dendrimers, a feasibility examination of dendrimer tagging of fingerprints has also been conducted.

KEYWORDS: forensic science, fingerprints, photoluminescence, semiconductor nanocrystal, quantum dot, nanoparticle, cadmium sulfide, dendrimer, time-resolved imaging, phase-resolved imaging

Because of its exceptional sensitivity and universality, i.e., applicability to a very wide range of articles of evidence, the detection of latent fingerprints via photoluminescence represents a new paradigm in criminalistics that has its beginning in 1976 (1). The essence of the approach, applicable to other evidence analyses also, has since seen much evolution (2). As with photoluminescence techniques quite generally, background fluorescence presents a problem that makes many articles of evidence intractable to fingerprint detection by the current routine photoluminescence procedures. Time-resolved imaging, which mitigates this problem, comes in two general categories. In time-domain techniques, also referred to as time-gated techniques, the excitation source is periodically turned on and off. The imaging device is synchronized with this light chopping such that it turns on during the chopping dark period, with a delay past the light cut-off such that the background fluorescence has already decayed by the time the imaging device turns on. The device turns off before the onset of the next light on period. One of the earliest implementations of this concept in luminescence lifetime measurement was by E. Bacquerel in 1871 (Ann Chim Phys 27: 539). The basic apparatus is referred to as a phosphoroscope. For fingerprint imaging, the underlying principle was first utilized in 1979 (3) but became feasible in a practical sense only in the late '80s (4) with the advent of microchannel plate image intensifiers and lanthanide-based fingerprint treatments. These treatments have since undergone considerable development (2). However, many problems remain to be solved in connection with fingerprint treatments that yield the required long luminescence lifetimes, with older fingerprints on porous surfaces in particular. Thus, more recent attention has begun to target frequency-domain time-resolved imaging, also referred to as phase-resolved imaging, as described for fingerprint detection already in 1995 (5). The exploitation of the underlying concept in photoluminescence lifetime measuring systems dates back to 1926 (6). One of us (ERM) developed in 1976 a phase-resolved fluorescence lifetime measuring system with lifetime resolution of better than 5 picoseconds (7), probably still a benchmark today. The basic concept has, regarding lifetimes, long been textbook material (8), and has in imaging systems been implemented in cell microscopy, for instance, since the early '90s (9). It calls for sinusoidal intensity modulation of the luminescence excitation source. The produced luminescence is then sinusoidal in intensity as well, but phase-delayed by an amount related to the luminescence lifetime through the equality of the tangent of the phase shift to the product of the photoluminescence lifetime and the modulation angular frequency. There is a concomitant well-defined decrease in modulation depth of the luminescence relative to the excitation with increasing luminescence lifetime. Thus, if the imaging device has its gain modulated with suitably adjusted phase with respect to the excitation, then fingerprint luminescence and background luminescence can be distinguished if their lifetimes differ sufficiently to be resolved. The fingerprint lifetime could in principle be shorter than that of the background for phase-resolved imaging, but short lifetimes usually are attended by low intensities. Whereas on is on practicality grounds limited in time-gated imaging to very long fingerprint luminescence lifetimes, typically milliseconds to microseconds, one can work in the nanosecond domain of molecular fluorescences with phase-resolved imaging (2). This broadens the range of potentially useful fingerprint treatments. Unfortunately, however, the obnoxious background fluorescences to be suppressed all too often have lifetimes (roughly in the 1–3 ns range) comparable to those of the photoluminescence fingerprint treatments currently in use, such as ninhydrin/zinc chloride and similar chemical treatments, and staining after cyanoacrylate ester fuming with rhodamine 6G and similar dyes (10). Thus, there is a need for a new fingerprint processing strategy that lends itself to the domain of phase-resolved imaging, if one wants to fully exploit background suppression, to tackle heretofore intractable evidence.

Currently, a frequency-domain lifetime imaging system for fingerprint detection is under development at Systems & Processes Engineering Corporation (SPEC, http://www.spec.com), 101 Sixth Street, Suite 200, Austin, TX 78701-2932. This is the first phase-resolved system to explicitly target the fingerprint field.

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Fingerprint Detection with Photoluminescent Semiconductor Nanocrystals

Phase-resolved detection tends to be most applicable when the luminescence lifetime of the latent fingerprint falls roughly in the 10–1000 nanosecond range. Lifetimes shorter than about 10 ns are too close to those of background fluorescences to be readily resolved. For lifetimes longer than microseconds, nothing is gained over time-gated fingerprint detection, which reached the maturity for practical implementation some time ago (11). Photoluminescent semiconductor nanocrystals, also referred to as nanocrystallites, quantum dots, nanoparticles, nanoclusters or nanocomposites, formed from compounds such as ZnS, CdS, CdSe, CdTe, InP, InAs, yield intense luminescences with lifetimes in the desired range. Moreover, the absorption and emission can be tailored by adjustment of the nanocrystal size. Nanocrystal capping, sometimes referred to also as derivatization or as functionalization, especially when the capping is with an organic compound (sometimes referred to as surfactant), involves covering the nanocrystal surface with a layer of material that can serve a number of purposes, namely passivation to optimize the luminescence efficiency and band width, serving as site for chemical attachment of conjugating ligands that also bind to target molecules to thereby fluorescently label them with the quantum dots, serving the labeling function themselves, serving to solubilize the nanocrystals, and preventing aggregation of the nanocrystals. As a result of these spectroscopic and capping virtues, nanocrystals are today receiving much attention in biochemistry (12,13). Application in a universal way has also been proposed (2,14) for fingerprint detection, with chemical and physical treatments envisioned, including labeling via conjugating ligands, analogous to approaches employed in some lanthanide-based fingerprint treatments (15), preferential adherence to or trapping in polymer voids (the cyanoacrylate polymer) or preferential adherence to the fingerprint residue itself, namely the approaches this article focuses on. In a perhaps more speculative vein, we envision incorporation into dendrimers, such as poly(amidoamine) dendrimers, which may preferentially attach to fingerprints via amine or carboxylate functional groups. This prospect is suggested by demonstration of the synthesis itself of various kinds of nanoparticles within dendrimer voids (16,17), which has brought nanoparticle-carrying dendrimers into recent limelight because of potential applications in catalysis, nanodevices, medicine, etc. In situ nanoparticle synthesis in voids of polymer aggregates and films has been reported as well (18,19), and may be applicable to the fingerprint context, perhaps with cyanoacrylate fuming. Dendrimers are polymeric macromolecules with repeat-units that branch out from a core, much like the branches of a tree. The dendrimers of interest in this article have a roughly spherical configuration of branches, referred to as a starburst topology (20).

In this communication, the reduction to practice of the general concept of luminescent semiconductor nanocrystal utilization for fingerprint detection is presented. As a preface to the fingerprint detection itself with the employed CdS nanocrystals, their pertinent photophysical properties are taken up to assess suitability for fingerprint work in concert with phase-resolved imaging techniques.

Spectroscopy of CdS Nanocrystals

We were fortunate to obtain from Professor John T. McDevitt (Department of Chemistry, University of Texas at Austin) a small sample of CdS nanocrystals, prepared in inverse micelles (21,22), and capped with dioctyl sulfosuccinate (sodium salt). Heptane is a preferred solvent to solubilize this kind of nanocrystal and we employed it, as well as hexane (actually a mixture of hexanes, $CH_3C_4H_8CH_3$). Solutions were prepared with quantum dot concentrations of milligrams/milliliter order. Figure 1 shows the absorption spectra of heptane and hexane solutions, the latter at lower concentration than the former. These spectra serve to determine the sizes of the nanocrystals (21), which we deduce to be of radii between 3 and 4 nm in heptane. Sizes between 5 and 10 nm in hexane are suggested by the red-shifted respective absorption spectrum of Fig. 1, but such a size change is difficult to understand, given that both heptane and hexane solutions were prepared from the same capped nanocrystal batch. Hexane solutions of the nanocrystals also show on near-ultraviolet excitation and visual inspection without any filter an easily noticeable luminescence blue-shift to orangish yellow from the reddish orange of heptane solutions. We do not as yet understand the cause of these effects (change in passivation?), but note the rather lower viscosity of hexane than of heptane, which might allow the former to better slip between the strands of the dioctyl succinate surfactant to access the nanocrystal surface. The luminescence spectral features of CdS (and other) nanocrystals are complex otherwise as well and depend on a number of factors. For instance, spectral shapes and intensities are excitation power dependent beyond the usual proportionality between excitation and emission intensity. Luminescence lifetimes are peculiarly excitation intensity dependent as well (at a given excitation wavelength), quite apart from variations with excitation color. These features (23,24) are quite different from what one is accustomed to in typical atomic or molecular lu-

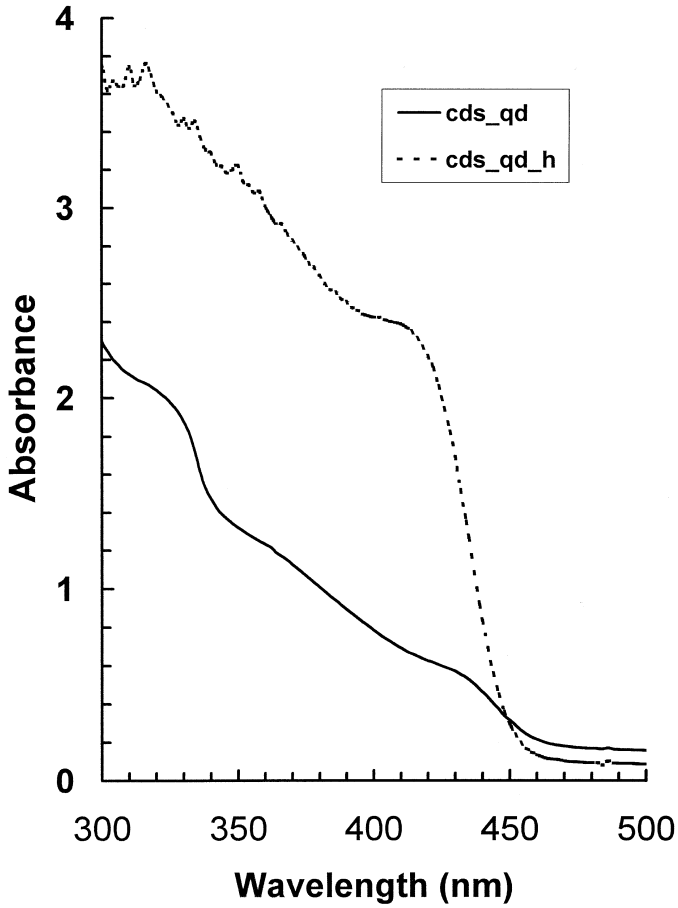

FIG. 1—*Absorption spectra of CdS nanocrystals in hexane (solid line, cds_qd) and heptane (dashed line, cds_qd_h). See text.*

minescence. They arise because the luminescence from the nanocrystal has several origins. An emission with short lifetime that is more pronounced toward the short-wavelength end of the emission spectrum in CdS is attributable to excitonic emission, referred to also as band-edge emission. This corresponds to the intrinsic semiconductor recombination process, and as such is lifetime-wise largely independent of wavelength, nanoparticle size and even temperature. A broad emission between roughly 350 and 800 nm, dominated by long-lifetime luminescence is due to traps at and near the nanocrystal surface. Since there are a number of trap states, luminescence lifetimes are very much a function of luminescence wavelength, temperature, and particle size, and they also are very susceptible to changes in the capping of the nanocrystal. Thus, in addition to the already cited solvent effect, one sees peculiar luminescence spectral changes with excitation power, as seen, for example, in Figs. 2A and B for heptane solution at room temperature. The shown spectra are technical spectra obtained with a photomultiplier tube of extended S 20 response and a monochromator equipped with a grating blazed at 500 nm. The sharp line features in the spectra are uninteresting plasma discharge lines from the Ar-laser (operating in the near-UV) that was used for sample excitation, excepting the strong line in Fig. 2 at 725 nm, which is a laser line reflected from the monochromator grating in second order. The excitation corresponding to the spectrum of Fig. 2A was well over an order of magnitude more intense than that corresponding to Fig. 2B. To obtain a reasonably large photon count, the monochromator slit widths were increased in the spectrum of Fig. 2B, as compared to Fig. 2A. The noteworthy feature in comparing the two spectra is the change in the relative intensities of the spectral feature in the 400–500 nm region (not counting the discharge lines) compared to the 500–700 nm feature. This is reminiscent of observed luminescence lifetime dependence on excitation power (23). At the laser power employed for fingerprint detection, corresponding to Fig. 2A, the broad band between 500 and 700 nm is more intense relative to the shorter wavelength shoulder than what is reported in Ref 23, not really surprisingly, given the capping difference. Since reductions in luminescence efficiencies are usually attended by shortening of luminescence lifetimes (increases in radiationless decay rates), we can anticipate from the spectrum of Fig. 2A that the long-lived luminescences in our case might, all else being equal, have longer lifetimes than those of Ref. 23, which they do, as we shall see shortly.

Because trap luminescences are strongly affected by excitation wavelength, intensity, capping, etc., one can use the literature only as a general guide as to what to expect. One needs to specifically determine the spectral features of the sample at hand, for the excitation condition employed, to optimize phase-resolved imaging. Accordingly, we measured luminescence lifetimes for our nanocrystal sample in heptane solution at room temperature (with a phase-resolved system that uses a HeCd laser operating at 325 nm with 15 mW of power). The lifetime corresponding to the total fluorescence was measured, rather than that corresponding to the luminescence at a particular wavelength. This was done by using a long-wavelength-pass filter that blocks the laser wavelength but transmits all longer ones. The rationale for this, rather than measuring lifetimes at a collection of specific wavelengths, is that the detection of a fingerprint usually involves the detection of the total fluorescence. Laser modulation frequencies ranged from 0.1 to 100

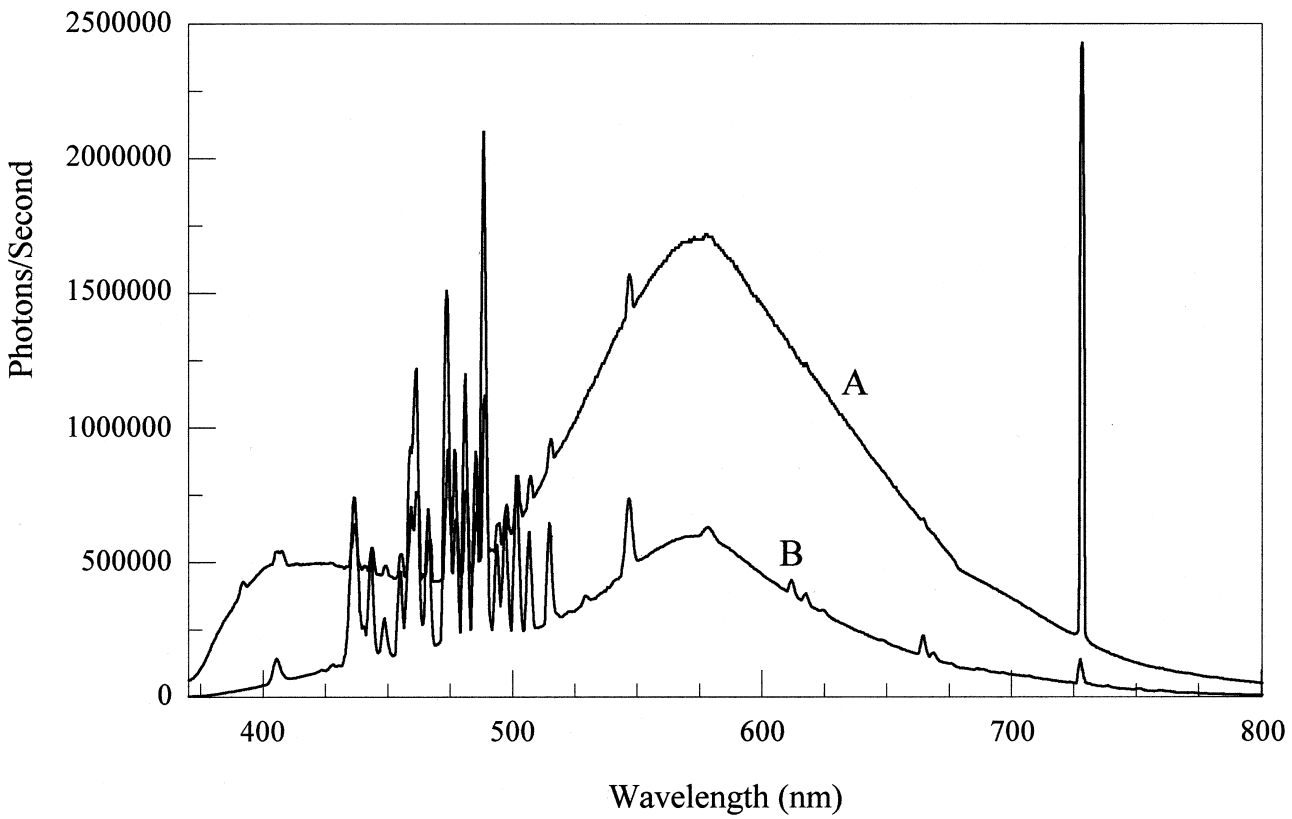

FIG. 2—*Room temperature fluorescence spectrum of heptane solution of CdS quantum dots capped with dioctyl sulfosuccinate under near-UV high power excitation (A) and low power excitation (B). See text.*

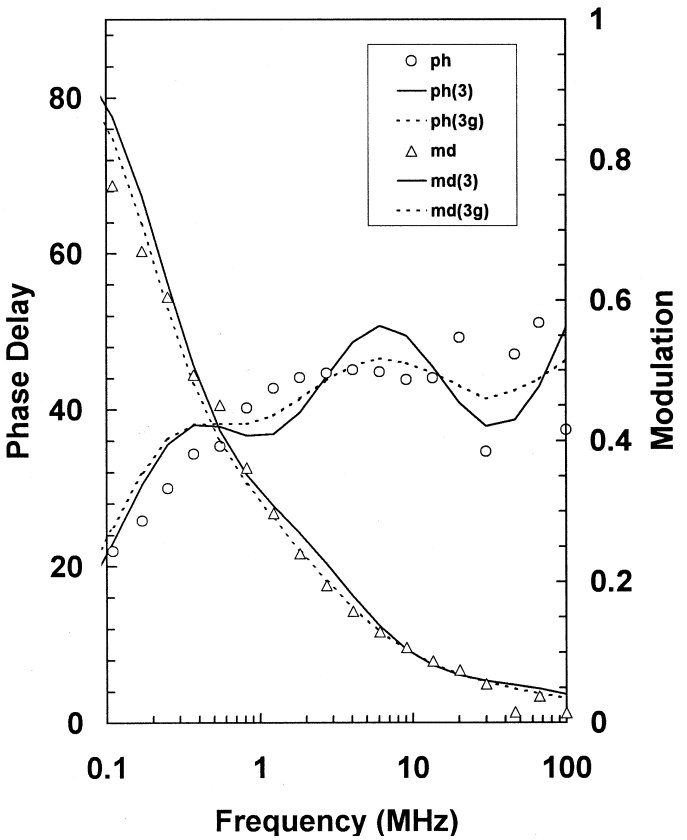

FIG. 3—*Phase-resolved spectroscopic data for CdS quantum dot luminescence lifetime determination. See text. ph = phase, md = modulation, (3) = fit with 3 discrete lifetimes, (3g) = fit with three Gaussian distributions.*

MHz. The corresponding phase delays and demodulation factors are shown in Fig. 3. Two analyses were performed, one assuming a superposition of discrete lifetime components and the other assuming a superposition of components made up each of Gaussian lifetime distributions. Results were similar. Three lifetime components were obtained. About 70% of the luminescence intensity corresponded to emission with lifetime of about 1000 ns, about 25% to emission with lifetime of about 70 ns, and about 5% to emission with lifetime of 0.54 ns. This latter is the excitonic emission and the obtained lifetime is in good agreement with the literature (23), as it should be. That the longest-lived emission lifetime reported by O'Neil, Mahron, and McLendon (23) is very roughly 200–300 ns, shorter than what we measure, is consistent with capping difference and the findings corresponding to Fig. 2A.

Our finding of long-lived luminescence that is intense enough to easily be visible to the naked eye indicates that the sample should be quite suitable for fingerprint processing and phase-resolved imaging over a wide range of modulation frequencies. We do not expect the solution lifetimes to change much on nanocrystal deposition on fingerprints, given the nanocrystal encapsulation.

Fingerprint Detection with CdS Nanocrystals

One of the most successful photoluminescence procedures for fingerprint detection consists of staining with fluorescent dye, typically after cyanoacrylate ester fuming (2). In our exploration of the feasibility of utilizing nanocrystals for fingerprint work, we used the above dioctyl sulfosuccinate capped CdS nanocrystals, dissolved in heptane or hexane. Sample fingerprints placed on aluminum foil and a soft drink can were fumed with cyanoacrylate ester and then immersed for times ranging from a few seconds to a few minutes in the nanocrystal solutions. The immersion times were not critical. The samples were then left to dry. Examination under an Ar-laser operating in the near-UV, which is a quite suitable excitation region, as seen from Fig. 1, revealed amply intense luminescence, easily visible. However, there was a generally heavy coverage of the immersed region, with no fingerprint ridge detail discernible. The samples were thus subjected to a gentle rinsing with hexane, to remove excess nanocrystal deposition, leaving behind fingerprint detail. Examples are shown in Fig. 4 for bare aluminum foil and the paint-covered metal of a soft drink can.

Unfumed fingerprints on metal, glass, and plastics, which normally are fumed, could not be developed. The reason is that hexane and heptane are rather good solvents for non-polar substances generally, and fingerprint lipids in particular. Thus, the very immersion of the article in the nanocrystal solution tends to obliterate ridge detail. In this respect, hexane is a more agressive solvent than heptane because of its substantially lower viscosity.

There are instances in which articles are customarily stained without prior fuming with cyanoacrylate ester. An example is that of fingerprints on the sticky side of adhesive tapes. Thus, we deposited prints on ordinary translucent Scotch™ tape and on black plastic electrical tape. There was strong adherence everywhere on the translucent tape, such that the heptane post-staining rinse (less agressive than the hexane rinse) did not do well in removing excess nanocrystals deposited everywhere on the tape, whereas hexane rinsing removed too much of the nanocrystal deposition. However, on the black electrical tape, fingerprints developed fairly readily with heptane solution staining and heptane rinsing. An example is shown in Fig. 5. The message to be gleaned is that there is a general need to optimize the solvent delivery system. If this is done, then the staining approach to nanocrystal utilization without the cyanoacrylate pretreatment should become more widely successful. The fingerprints of Figs. 4 and 5 were obtained with ordinary photography, rather than time-gated or phase-resolved imaging.

Further studies are underway, in terms of solvent optimization, including solubilization in polar solvents, in terms of the semiconductor material, where CdSe is of particular interest because it is nicely compatible with excitation in the blue-green, in terms of capping, whether with organic functionalization or inorganic encapsulation (e.g., ZnS or silica), in terms of choice of conjugating ligands for selective chemical labeling, as suggested by the example of mercaptoacetic acid in concert with ZnS capping (13), and in terms of the earlier-discussed dendrimer application. The success we have already obtained, even if only with a sparse portfolio of procedures, as dictated by sparse nanocrystal accessibility to us in quantity and kind, is encouraging, particularly regarding the cyanoacrylate/nanocrystal fingerprint treatment. It leads us to believe, on the basis of flexibility in size and semiconductor composition, capping, ligand attachment, and, finally, exceptional suitability for background suppression due to long-lived luminescence of high intensity, that nanocrystals are destined to yield major advances in fingerprint detection. It is our understanding that commercialization of a range of nanocrystal kinds is in the offing.

Fingerprint Tagging with Dendrimers

In situ assembly of nanoparticles in voids within dendrimers has been demonstrated, and shows much promise in various fields. The

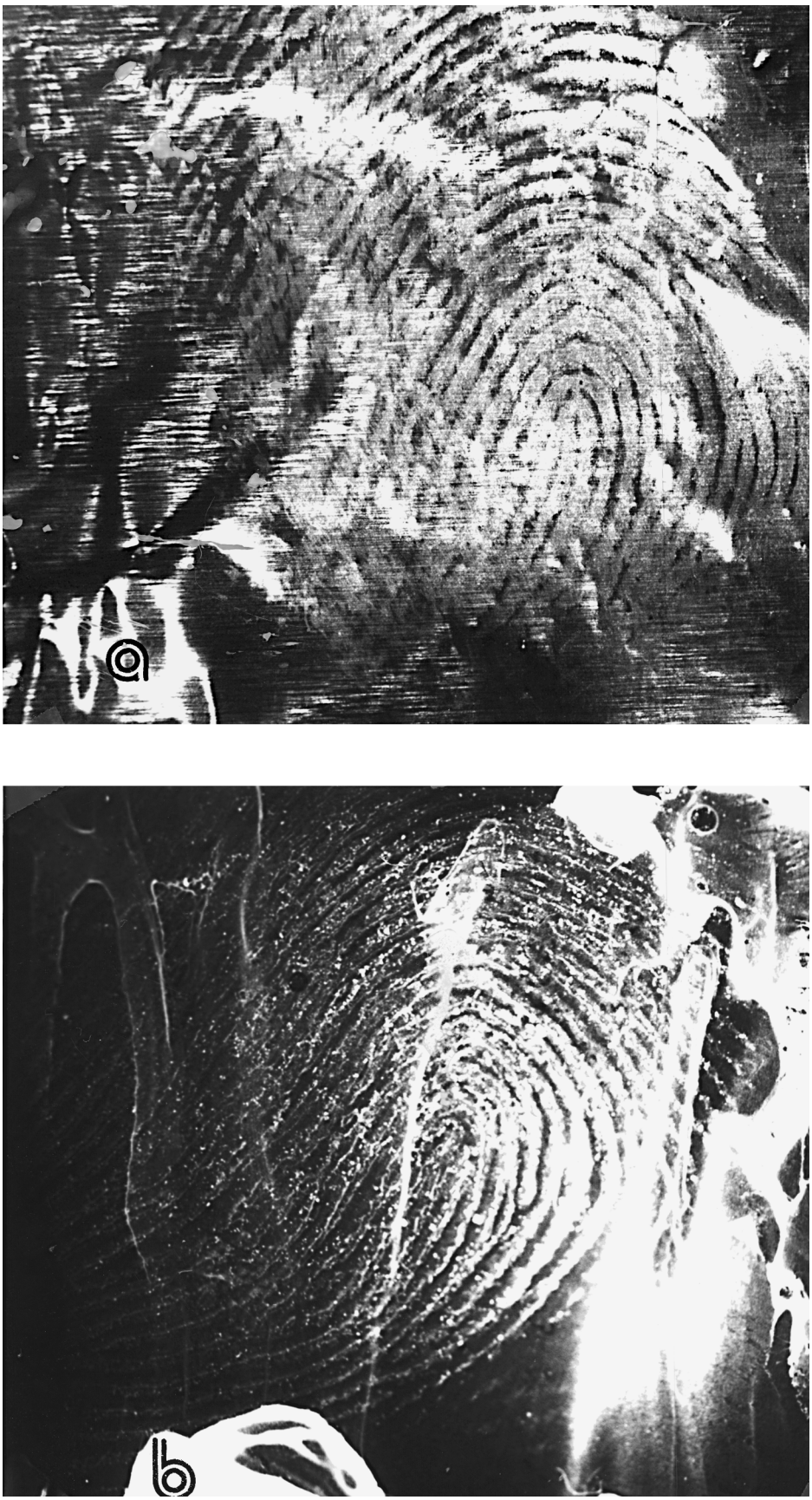

FIG. 4—*Photograph of fingerprints developed by cyanoacrylate/CdS nanocrystal staining on aluminum foil (a) and soft drink can (b).*

FIG. 5—*Photograph of fingerprint on sticky side of black plastic electrical tape directly developed by staining with CdS nanocrystals.*

preparation of a CdS/dendrimer nanocomposite that displays intense photoluminescence has recently been reported as well (25). Dendrimers utilized in these investigations are Starburst® (PAMAM) dendrimers, commercially available from Aldrich, that are functionalized with amino, hydroxy or carboxylate groups, thus offering the prospect of serving as taggants in addition to being nanocomposites. Accordingly, we have carried out a preliminary investigation aimed at determining whether it is at all worthwhile to contemplate fingerprint tagging with dendrimers. In our feasibility examination, we chose Starburst^{R} Generation 4 dendrimer, which contains surface amine groups. The choice was made with potential reaction with carboxylic acids, esters, perhaps glycerides in mind, to form amide linkages. Since OH is a poor leaving group, the reaction with carboxylic acid usually involves first conversion to an ester via carbodiimide. In our preliminary work, we kept in mind the delicacy of fingerprints, i.e., their potential obliteration by solvents. We thus confined ourselves to simply applying the dendrimers, which come in methanol solution, by spotting onto fingerprints and letting dry, with and without heating. Whether in solution or dried spots, the dendrimers are colorless and non-luminescent. Thus, it was necessary to find a way to visualize them. We chose staining with rhodamine 6G or resorufin because these molecules readily penetrate polymers to deposit in voids. Both are luminescent under blue-green excitation. The rhodamine 6G fluorescence is typically greenish yellow, which is not optimal because the color is rather similar to inherent fingerprint fluorescence, whereas the resorufin fluorescence is orange, very distinct from inherent fingerprint fluorescence.

The dendrimer methanol solution is rather viscous and forms a thick, tacky coating on drying, on the polyethylene and aluminum foil we used as substrates onto which fingerprints were deposited. Prolonged heating (56°C) causes it to become brownish. Nothing to speak of was gained by the heating, which was thus discontinued in our fingerprint treatment procedure. After the room temperature drying, spotted items were immersed in (about 10^{-3} molar) methanol solution of either rhodamine 6G or resorufin for times from about 10 seconds to several minutes, and then left to dry again. The immersion time was found not to be important. Finally, thus processed samples were subjected to a very vigorous rinse with methanol, to remove the thick coating covering the spotted region of the sample. With polyethylene, fingerprints tended to be washed away. We thus mostly focused on fingerprints deposited on aluminum foil, which has the added virtue of being non-luminescent under blue-green (Ar-laser) excitation. Since rhodamine 6G, for instance, is well known to stain fingerprint residue itself, it is necessary for dye staining of fingerprints to differ in effectiveness from dendrimer dye staining if any visualization of the latter is to be achieved. This is a matter of penetration into or attachment to the dendrimer, and retention there upon the subsequent methanol rinse, in comparison to the binding to and retention on the fingerprint residue itself. Spotted fingerprints stained with rhodamine 6G tended to show uniform fluorescence with reasonably crisp ridge detail, whereas unspotted prints usually showed spotty development with considerable obliteration of ridge detail, but with typically more intense fluorescence than spotted prints. With too little rinsing, intensely luminescent residual coating would be apparent and with excessive rinsing, fingerprints would be obliterated. The situation is complicated further by the inherent fingerprint fluorescence (of the same color as that of the rhodamine). Thus, we eventually settled

FIG. 6—*Tip of fingerprint on aluminum foil, spotted with dendrimer and stained with resorufin. See text.*

on spotting part of a fingerprint and leaving another part bare, to ensure that both portions would be stained and rinsed in the same way. With resorufin staining, spotted portions of fingerprints showed orange-luminescing ridge detail whereas the corresponding unspotted portions generally had washed-out detail with weaker fluorescence, indicative of comparatively less effective staining of fingerprint residue itself by the resorufin. An example is shown in Fig. 6, where the tip only of the fingerprint was spotted. Clearly, there is preferential dendrimer attachment to the spotted fingerprint portion. Whether this is chemical bonding or adherence by a physical process is not clear. We suspect the former, given the vigor of the employed methanol rinsing. In any event, our results suggest that a detailed investigation of fingerprint tagging with dendrimers for purposes of subsequent processing that involves photoluminescent semiconductor nanocomposites should be worthwhile.

Note Added in Review

Since the submission of this article, we have succeeded in developing latent fingerprints with CdS/dendrimer nanocomposites. This makes it interesting to investigate whether it is best to label fingerprints first with dendrimer and then incorporate the CdS or whether one should first prepare the CdS/dendrimer nanocomposite and then label the fingerprint with it. Furthermore, optimization studies are called for regarding dendrimer size, absolute and relative CdS/dendrimer concentrations, solvent systems, fingerprint tagging chemistry, photoluminescence lifetime, etc. These studies are underway and will be reported on in future publication.

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