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Crosslinkable Carbazolyl-Containing Molecular Glasses for Electrophotography

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The synthesis and properties of well defined carbazolyl-containing molecular glasses is reported. They were prepared by the nucleophilic opening of the oxirane ring of 1,3-di(carbazol-9-yl)-2-propanol, 1-(carbazol-9-yl)-3-diphenylamino-2-propanol or 1-(3,6-dibromocarbazol-9-yl)-3-(carbazol-9-yl)-2-propanol glycidyl ethers with 1,3-benzenediol in the presence of triethylamine. The electrophotographic parameters of undoped films of the molecular glasses and of those doped with difluoroboron-1,3-bis(4-methoxyphenyl)-1,3-propanedionate have been studied. The hole drift moblities measured by the time of flight technique in the molecular glasses containing carbazole and diphenylamino groups reach 10^{-4} cm^2 V^{-1} s^{-1} at an electric field of 10^6 V cm^{-1}. The ionization potentials measured by electron photoemission method in air are close to those reported for the other organic photoconductors containing electronically isolated carbazole moieties.

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Introduction

Since the discovery of photoconductivity in poly(9-vinylcarbazole) (PVK)[1] derivatives of carbazole became the subject of numerous investigations for applications in electrophotography, light emitting diodes, photorefractive materials.[2] The polymers and oligomers of N-(2,3-epoxypropyl)carbazole (PEPK) are among the most widely studied photoconductive materials.[3] High-molar-mass PEPK has been prepared in an analogous reaction of poly(epihalohydrin) with carbazole.[4] It exhibits one of the highest hole mobilities among the photoconductive polymers with pendant photoactive groups.[5] Low molar mass PEPK prepared by ionic polymerization of the monomer has been used for electrophotographic microfilming.[6] Oligomeric PEPK possesses low glass transition temperature (T_g) and relatively good film-forming properties. Unfortunately PEPK, like all polymeric materials, exhibits polydispersity with respect to molar mass and end groups, and the possibilities of its purification are limited. For the application in electrophotography in 1980's we have started to synthesize well defined low-molar-mass photoconductive compounds which are able to form amorphous films on substrates, including flexible ones.[7,8] Low-molar-mass compounds capable of existing in amorphous state have been called molecular glasses or amorphous molecular materials.[9] We prepare such compounds by the reactions of oxiranes containing photoconductive groups with different bifunctional compounds such as aromatic diols, dimercapto compounds, and derivatives of aniline. Previously we reported on well defined amorphous organic photoconductors prepared by the reaction 1,3-di(carbazol-9-yl)-2-propanol glycidyl ether (1a) and 1-(carbazol-9-yl)-3-diphenylamino-2-propanol glycidyl ether (1b) with different bifunctional compounds as the linking agents.[10] Despite the lower concentration of chromophores the electrophotographic photosensitivity some of these organic photoconductors doped with the sensitizer difluoroboron-1,3-bis(4-butoxyphenyl)-1,3-propanedionate (DBBFP) exceeds the photosensitivity of PEPK doped with the same sensitizer.

In this article we report on the synthesis, characterization and photoconductive properties of amorphous molecular materials prepared by the reaction of 1,3-benzenediol (resorcinol) with 1a,b and 1-(3,6-dibromocarbazol-9-yl)-3-(carbazol-9-yl)-2-propanol glycidyl ether (1c) in the presence of triethylamine (TEA). The resulting compounds contain hydroxy groups

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which make them crosslinkable using, for example, polyisocianates. Such crosslinked systems enable us to prepare electrophotographic photoreceptors with high solvent resistance and good mechanical properties.[11]

Experimental

Measurement

The ^{1}H NMR spectra were determined in deuterochloroform ($CDCl_3$) using a Hitachi R-22 (90 MHz) spectrometer; the chemical shifts are expressed in ppm, downfield from hexamethyldisiloxane (HMDS), used as internal standard. The symbols *s, d, m* are used for singlet, doublet and multiplet respectively. Coupling constants (*J*) in Hz are presented. The ^{13}C spectra were recorded on a Tesla BS 567A (25. 142 MHz) spectrometer with tetramethylsilane (TMS) as the internal standard. The IR spectra were taken for samples in KBr pellets on a UR-20 spectrometer and selected bands are presented. The UV spectra were recorded on a Spectronic Genesys 8 spectrometer. The fluorescence spectra were recorded on a Hitachi MPF-4 spectrofluorometer in acetonitrile; 10^{-4} M solutions of the investigated materials in microcells with internal widths of 1 mm and 10 mm were used. Differential scanning calorimetry measurements were recorded on a Mettler DSC 30 calorimeter at a scan rate of 10 K/min.

The samples for the measurements of electrophotographic photosensitivity were prepared by casting the solutions of 2a-c in toluene on the glass substrates primed with an SnO_2 layer. The thickness of the amorphous films of 2a-c varied in the range of 3 – 5 μm. Parallel layers of 2a-c containing 1 wt. % of DBBFP, which is regarded as one of the most effective sensitizers for carbazolyl-containing organic photoconductors,[12] were prepared. For the photosensitivity measurements the layers were charged by corona up to 600–800 V and were illuminated with monochromatic light, the intensity of which was 10^{17} quanta/m^2·s. The photosensitivity was calculated from the time of half decay of the initial potential.

The residual potential U_R was found as a potential value at the time $t = 10 \cdot U_0/(dU/dt)_{t=0}$ during illumination. Here U_0 is the potential value at the beginning of illumination; $(dU/dt)_{t=0}$ is the initial photodischarge speed. The corresponding exposure is $U_0/(dU/dt)_{t=0} \cdot 10^{18}$ quanta/m^2·s.

The photogeneration quantum efficiency for incident light was calculated according to formula

$$\beta = \frac{\varepsilon\varepsilon_0 hc}{eL_\lambda \lambda d(1-R)} \cdot \left(\frac{dU}{dt}\right)_{t=0},$$

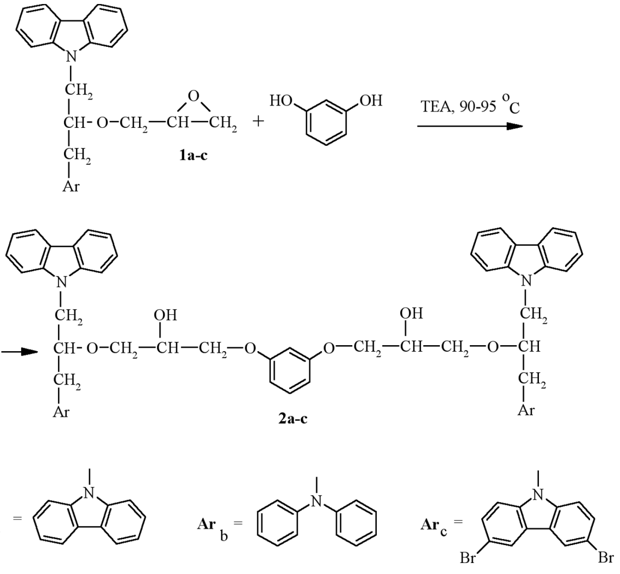
where ε is the layer dielectric constant, *L*λ is the light intensity, λ is the light wavelength, *U* is the layer potential, *R* is the reflection coefficient, and *d* is the layer thickness. The ratio β / ε is plotted as the ordinate, since ε was not measured experimentally.

The hole drift mobility was measured by the time of flight technique. [13] An electric field was created by positive corona charging. The charge carriers were generated at the layer surface by illumination with pulses from a nitrogen laser (pulse duration was 2 ns, wavelength 337 nm). The layer surface potential decrease as a result of pulse illumination was up to 1–5 % of the initial potential before illumination. A capacitance probe connected to the wide frequency band electrometer measured the speed of the surface potential decrease d*U*/d*t*. The transit time t_t was determined by the kink on the curve of the d*U*/d*t* transient in double logarithmic scale. The drift mobility was calculated from the formula $\mu = d^2/U_0 t_t$, where d is the layer thickness, and U_0 is the surface potential at the moment of illumination.

The samples for the ionization potential measurement were prepared by dissolving materials in THF. They were coating on Al plates pre-coated with ~0. 5 μm thick poly(methylmethacrylate)-co-(methacrylic acid) adhesive layer. The thickness of the transporting material layer was 0. 5 – 1 μm. The ionization potential I_p was measured by electron photoemission in air, similar to the method described in Ref. 14. Usually such photoemission experiments are carried out in vacuum, and high vacuum is one of the main requirements for these measurements.[15,16] If the vacuum is not high enough the sample surface oxidation and gas adsorption influence the measurement results. In our case, however, the organic materials investigated are stable enough towards oxygen so that the measurements may be carried out in air.

The samples were illuminated with monochromatic light from a quartz monochromator with a deuterium lamp. The power of the incident light beam was 2 – 5 × 10^{-8} W. A negative voltage of –300 V was applied to the sample substrate. The counterelectrode with a 4. 5 × 15 mm^2 slit for illumination was placed at an 8 mm distance from the sample surface. The counter electrode was connected to the input of the BK2-16 type electrometer, working in the open impute regime, for photocurrent measurement. The 10^{-15} – 10^{-12} A photocurrent flowed in the circuit under illumination. The photocurrent *I* was found to be strongly dependent on the incident light photon energy hν, and an $I^{0.5} = f(h\nu)$ dependence was plotted. Usually the dependence of the photocurrent on incident light quanta energy is adequately described by the linear relationship between $I^{0.5}$ and *h*ν near the threshold.[14–16] The linear region of this dependence was extrapolated to the hν axis and I_p value was determined as the photon energy at the intercept point.

Materials



Scheme 1. Synthesis route of carbazolyl-containing molecular glasses 2a-c.

The general synthesis route to the branched carbazolyl-containing molecular glasses 2a-c is shown in scheme 1. Starting glycidyl ethers 1a-c were obtained according to our earlier methods, described in Ref. 17, while the sensitizer DBBFP was prepared according to the procedure described in Ref. 12.

Synthesis of 1,3-bis[6-(carbazol-9-yl)-5-(carbazol-9-methyl)-2-hydroxy-4-oxahexyloxy]benzene (2a).

22. 3 g (50 mmol) of 1a and 2. 2 g (20 mmol) of resorcinol were dissolved in 50 mL of chlorobenzene and 0. 7 mL (5 mmol) TEA were added. The mixture was stirred at 90 – 95°C until the resorcinol and its monosubstituted derivative disappeared (33 h). The course of the reaction was monitored by thin layer chromatography on Silufol UV-254 (Kavalier) plates using ether-*n*-hexane (2:1) as the eluent. At the end of the reaction chlorobenzene and TEA were distilled off and the product 2a was isolated by subjecting the residue to chromatography (silica gel L 40/100, Chemapol) using ether-*n*-hexane (2:1) as the eluent. After removal of the eluents, the 20% solution of the oily residue in toluene was poured with intensively stirring into a tenfold excess of *n*-hexane; 14. 0 g (69. 8%) of a white powder was obtained. T_g = 76°C. ^{1}H NMR spectrum: 1. 58 (2H, s, OH); 3. 20 (10H, m, O-CH_2CHCH_2-O); 4. 37 (10H, m, N-CH_2CHCH_2-N); 6. 11 (3H, m, 2-H, 4-H, 6-H *m*-Ph); 7. 15 (25H, m, 5-H *m*-Ph, Ht); 8. 01 (8H, d, 4-H, 5-H Ht, J=7. 0). ^{13}C NMR spectrum: 45. 53; 67. 76; 69. 10; 72. 46; 78. 18; 101. 52; 106. 33; 109. 51; 119. 04; 120. 32; 122. 29; 125. 72; 129. 60; 140. 40; 157. 44. IR spectrum, cm^{-1}: 3600-3300 (OH); 3060, 3030 (CH_{arom}), 2940, 2887 (CH_{aliph}), 1125, 1155 (C-O-C). Found, %: C 78. 82; H 5. 68; N 5. 38. $C_{66}H_{58}N_4O_6$. Calculated, %: C 79. 02; H 5. 83; N 5. 58.

Synthesis of 1,3-bis[6-(carbazol-9-yl)-5-(diphenylamino-N-methyl)-2-hydroxy-4-oxahexyl-oxy]benzene (2b)

2b was prepared and isolated as described for 2a, except that instead of 1a 22. 4 g (50 mmol) of 1b were used. The reaction time was 48 h. Yield was 14. 8 g (73. 3%), T_g = 51. 5°C. ^{1}H NMR spectrum: 1. 77 (2H, s, OH), 2. 88-3. 71 (10H, m, O- CH_2CHCH_2-O); 3. 71-4. 52 (10H, m, N-CH_2CHCH_2-N); 6. 08 (3H, m, 2-H, 4-H, 6-H *m*-Ph); 6. 66-7. 55 (33H, m, 5-H *m*-Ph, Ht, Ph); 7. 93 (4H, d, 4-H, 5-H Ht, J=7. 0). ^{13}C NMR spectrum: 45. 72; 54. 76; 68. 28 (68. 87), 72. 24; 77. 65; 79. 14; 101. 50; 107. 01; 108. 69; 119. 26; 120. 41; 120. 97; 121. 80; 122. 92; 125. 75; 129. 37; 129. 60; 140. 43; 147. 90; 157. 44. IR spectrum, cm^{-1}: 3600-3300 (OH), 3060, 3026 (CH_{arom}), 2936, 2884 (CH_{aliph}), 1225, 1157 (C-O-C). Found, %: C 78. 68; H 6. 10; N 5. 36. $C_{66}H_{62}N_4O_6$. Calculated, %: C 78. 70; H 6. 20; N 5. 56.

Synthesis of 1,3-bis[5-(3,6-dibromocarbazol-9-methyl)-6-(carbazol-9-yl)-2-hydroxy-4-oxa-hexyloxy]benzene (2c)

2c was obtained and isolated as described for 2a, except that instead of 1a 30. 2 g (50 mmol) of 1c were used. The reaction time was 26 h. Yield was 18. 5 g (70. 1%), T_g=96°C. ^{1}H NMR spectrum: 1. 61 (2H, s, OH), 2. 75-3. 72 (10H, m, O-CH_2CHCH_2-O); 3. 72-4. 75 (10H, m, N-CH_2CHCH_2-N); 6. 0 (3H, m, 2-H, 4-H, 6-H *m*-Ph); 6. 57-7. 62 (21H, m, 5-H *m*-Ph, Ht); 7. 62-8. 12 (8H, m, 4-H, 5-H Ht). ^{13}C NMR spectrum: 44. 49; 45. 31; 67. 90; 68. 72; 71. 82; 77. 67; 102. 04; 105. 96(106. 56); 108. 35; 109. 81; 112. 06; 119. 44; 120. 45; 122. 77; 122. 88; 122. 92; 125. 87; 128. 74; 129. 86; 138. 57; 139. 34; 156. 83. IR spectrum, cm^{-1}: 3600-3300 (OH), 3060, 3030 (CH_{arom}), 2940, 2887 (CH_{aliph}), 1125, 1060 C-O-C). Found, %: C 60. 0; H 3. 93; Br 24. 21; N 4. 33. $C_{66}H_{54}Br_4N_4O_6$. Calculated, %: C 60. 11; H 4. 13; Br 24. 23; N 4. 25.

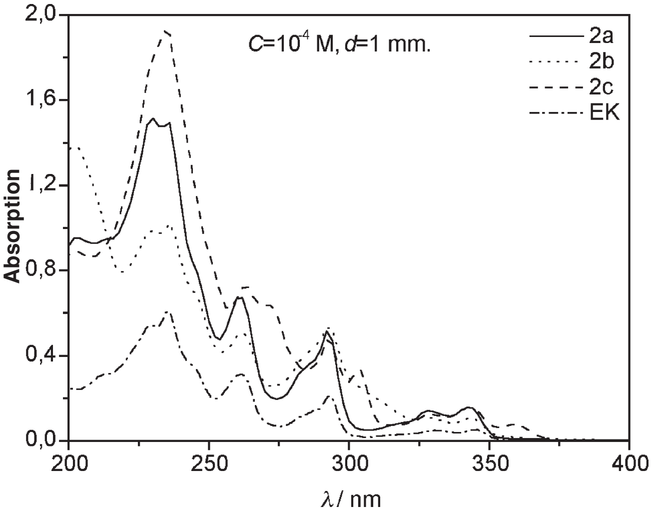


Figure 1. Ultraviolet absorption spectra of the solutions of 2a-c and N-ethylcarbazole in CH_3CN (c = 10^{-4} M).

Results and Discussion

The nucleophilic opening of the oxirane rings of glicidyl ethers 1a-c by heating with 1,3-benzenediol (molar ratio 2:1) at 90–95°C in the presence of TEA gave 1,3-bis[6-(carbazol-9-yl)-5-(carbazol-9-methyl)-2-hydroxy-4-oxahexyloxy]benzene (2a), 1,3-bis[6-(carbazol-9-yl)-5-(diphenylamino-N-methyl)-2-hydroxy-4-oxahexyloxy]benzene (2b) and 1,3-bis [5-(3,6-dibromocarbazol-9-methyl)-6-(carbazol-9-yl)-2-hydroxy-4-oxahexyloxy]benzene (2c). The compounds 2a-c were isolated by column chromatography followed by precipitation with a large excess of hexane. Isolated by such a procedure 2a-c are amorphous compounds. All our attempts to crystallize them were unsuccessful. X-ray diffraction patterns of these compounds show only broad halos. Such high morphological stability of these glasses can apparently be explained by existance of several diastereoisomers, the possibility of intermolecular hydrogen bonding and flexibility of the aliphatic linking chains. The glass transition temperatures (T_g) of the compounds 2a-c established by differential scaning calorimetric (DSC) are 76°C, 51. 5°C, and 96°C. The compound 2b containing diphenylamino groups, which are more flexible than carbazolyl groups, shows lower T_g than the compound 2a, while the compound 2c containing heavy bromine atoms exhibits higher T_g than the compound 2a.

The absorption spectra of dilute CH_3CN solutions of 2a-c are shown in Fig. 1. The spectra of N-ethylcarbazole (EK) are given for the comparison. The optical absorption spectra of the molecular glasses **2a-c** are very similar to that of EK and show the bands typical of N-substituted carbazole chromophore due to $n\rightarrow\sigma^*$, $\pi\rightarrow\pi^*$ and $n\rightarrow\pi^*$ transitions. The uv spectrum of **2c** is shifted to longer wavelengths apparently due to the presence of the bromine atoms. Figure 2 shows spectral distribution of photosensitivity (*S*) of amorphous films of the pure compounds 2a-c and of those doped with 1 wt. % of DBBFP. The values of photosensitivity and relative residual potential at the characteristic wavelengths

TABLE I. Photosensitivity *S* and Relative Residual Potentials U_R/U_0 of the Films of Undoped and Those Doped with 1 wt.% DBBFP

Layer composition	Thickness (μm)	U_0 (V)	$S_{1/2}$ (m^2/J) 295 nm	U_R/U_0 295 nm	$S_{1/2}$ (m^2/J) 345 nm	$S_{1/2}$ (m^2/J) 425 nm	U_R/U_0 425 nm
2a	4.8	730	7.5	0.176	10	—	—
2a+DBBFP	4.2	640	28	0.2	28	20	0.10
2b	6.1	745	4.6	0.14	4.4	—	—
2b+DBBFP	6.8	630	27	0.144	29	25	0.084
2c	5.2	870	2.3	0.125	1.8	—	—
2c+DBBFP	5.4	695	46	0.108	50.5	54	0.069

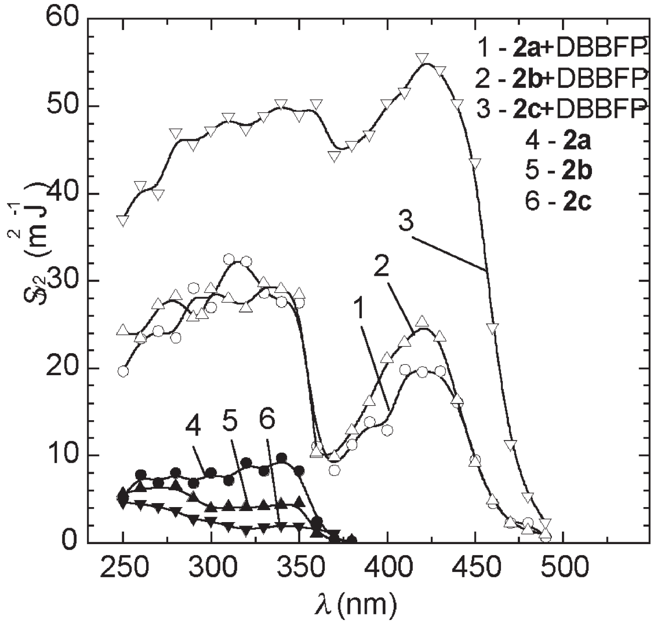


Figure 2. Spectral photosensitivity of the electrophotographic layers of the molecular glasses 2a-c.

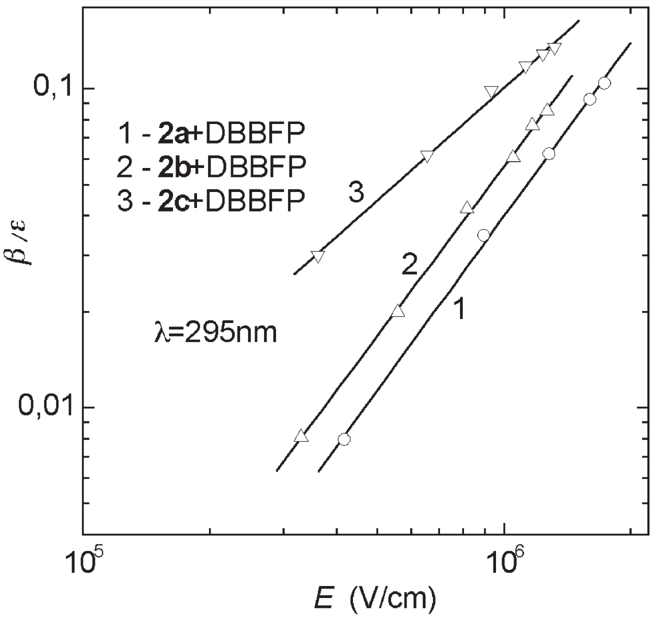


Figure 3. Field dependencies of the photogeneration quantum efficiency in sensitized layers of the molecular glasses 2a-c.

of the films are summarised in Table I. Pure molecular glasses 2a-c exhibit low photosensitivity, even in the UV region of the spectrum. Doping with DBBFP increases intrinsic photosensitivity in the UV region and induces photosensitivity in the visible region with a maximum at 425 nm. The lowest intrinsic photosensitivity is observed for the compound 2c, containing 3,6-dibromocarbazolyl groups. However, after doping with DBBFP, 2c exhibits the highest photosensitivity. For the doped 2c the photosensitivity observed in the visible region is higher than that observed in the UV region. These observations are consistent with the previously reported results.[18] Undoped brominated PEPK exhibited lower photosensitivity than PEPK itself, however the brominated oligomer was much more efficiently sensitized by pyrylium salts. Lower intrinsic photosensitivity of the brominated carbazolyl-containing photoconductors compared to that of the unbrominated analogues may be explained by the loss of some fraction of the singlet excitons by intersystem crossing, under the influence of heavy bromine atoms (see below). High sensitizing efficiency of DBBFP and pyrylium salts with respect to brominated carbazolyl-containing photoconductors is caused by high quantum efficiency of the photogeneration of electron-hole pairs.[19]

The comparison of photosensitivity of 2a and 2b show that the change of two carbazolyl groups by diphenylamino groups in the molecules of molecular glasses has almost no influence on their photosensitivity both in doped and in undoped state.

Figure 3 shows the electric field dependencies of charge carrier photogeneration quantum yield for the films of 2a-c doped with 1 wt. % of DBBFP. For all the compounds studied photogeneration quantum yield is strongly field dependent. These dependencies may be approximated by a power law function, $\beta \sim E^n$, with n = 1. 8 for 2a and 2b while in the case of 2c n=1. 2. The highest photogeneration quantum yield is observed for the doped compound 2c, which contains 3,6-dibromocarbazolyl groups. This observation shows that high photosensitivity in the sensitized 2c is mainly due to the high photogeneration efficiency. Over the entire range of electric field studied the doped molecular glass 2b shows a higher photogeneration efficiency and lower relative residual potential than the doped compound 2a. This observation can be explained by a lower concentration of charge carrier traps in compound 2b, owing to the presence of diphenylamino groups, which are more flexible than carbazolyl groups.

Examples of the surface potential transients observed during mobility measurements are shown in Fig. 4, and the plots of hole drift mobilities of the molecular glasses 2a-c versus electric field are shown in Fig. 5. In all cases the hole drift mobilities in the doped with DBBFP layers

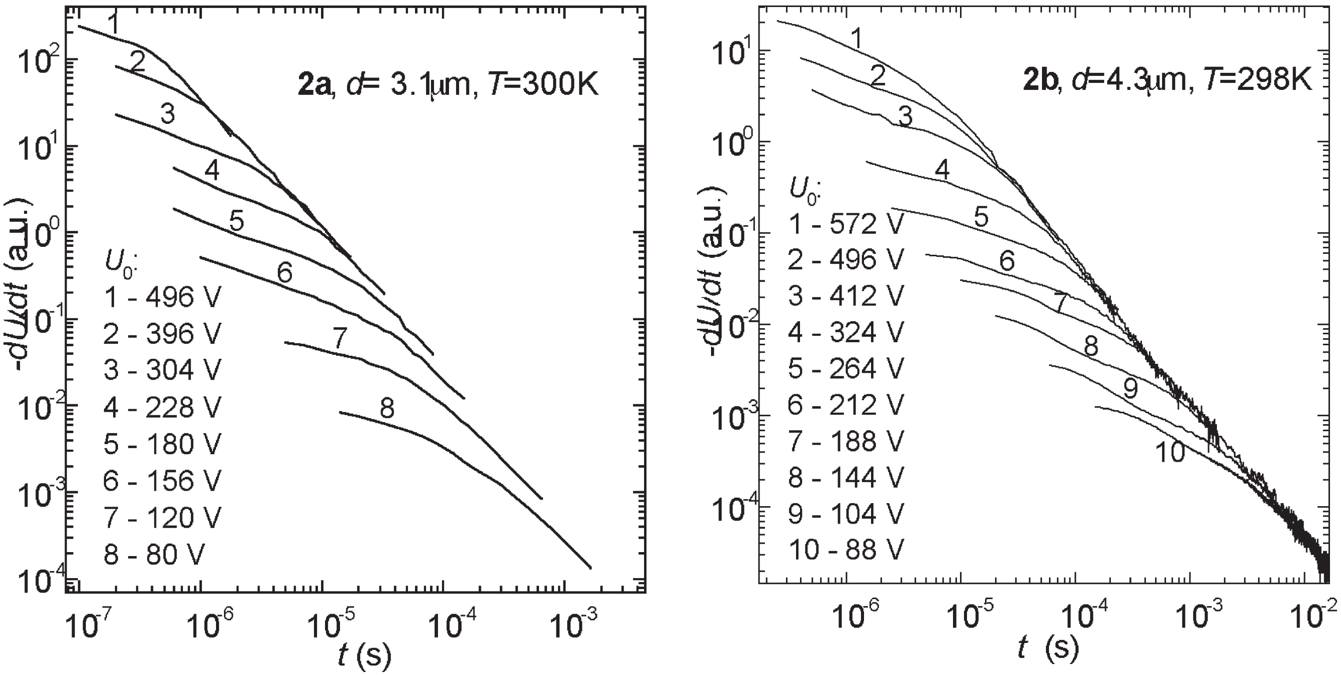


Figure 4. The surface potential transients recorded during mobility measurements for the layers of 2a (left) and 2b (right).

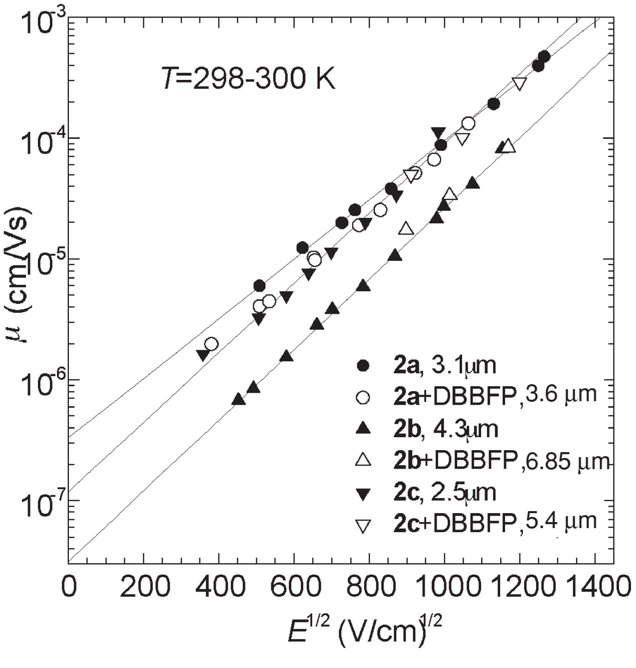


Figure 5. Field dependencies of the hole drift mobilities in the undoped and doped with 1 wt. % of DBBFP layers of the

2a-c are very close to the values observed for the undoped samples. The mobilities in 2a and 2c are very close, however they are considerably lower in the samples of 2b. This observation can apparently be explained by the higher energetic disorder in the later photoconductor.

The hole drift mobilities in the amorphous films of 2a and 2c reaches 10^{-4} cm^2 V^{-1} s^{-1} at an electric field of 10^6 V cm^{-1}. This is a rather high mobility, keeping in mind that carbazole chromophores are electronically isolated in these molecules. Similar, or even lower hole mobilities have been observed in many molecular glasses consisting of π-conjugated molecules.[9] Apparently total conjugation of molecules is not an obligatory precondition for effective charge transport in molecular glasses. These results may be compared to the mobilities in the other organic materials containing carbazole moieties such as PVK[20] or oligomeric PEPK.[21] According to Gill[20] the hole mobility in PVK at $5 \cdot 10^5$ V/cm electric field and 295 K temperature is ca. 1.5×10^{-6} cm^2/V·s. In the case of oligomeric PEPK the hole mobility is ca. 1.7×10^{-5} cm^2/V·s under similar conditions.[21] The hole mobilities in the molecular glasses 2a and 2c are about one order of magnitude higher than in PVK and somewhat higher than in PEPK. This may be due to more favorable spatial arrangement of the conjugated pendant groups in these soft structured materials, compared with the rigid chain polymer PVK. The important advantage of the molecular glasses 2a-c with respect to PVK is the lack of excimer forming sites in these compounds. This fact can also explain higher charge mobilities in these materials as compared with PVK. Figure 6 shows fluorescence spectra of dilute solutions of 2a-c. For comparison, the spectrum of a dilute solution of EK, is presented. The spectra of 2a,b are almost identical to that of EK and only the structured monomer fluorescence is observed. The fluorescence of a dilute solution of 2c is markedly quenched relative to that of the solutions of 2a,b at the same concentration, due to the presence of the heavy bromine atoms.

Charge-transporting materials are usually crystalline compounds and are used for layer preparation in compositions based on a polymeric binder such as polycarbonate. A number of such compositions have been studied.[22] The hole mobility values in the range of $(0.87 - 30) \times 10^{-6}$ cm^2/V·s at an electric field of 3×10^5 V/cm are reported. Mobilities in the molecular glasses described in this presentation are in the same range, but the mobility field dependencies is, as a rule, are stronger in the molecular glasses.

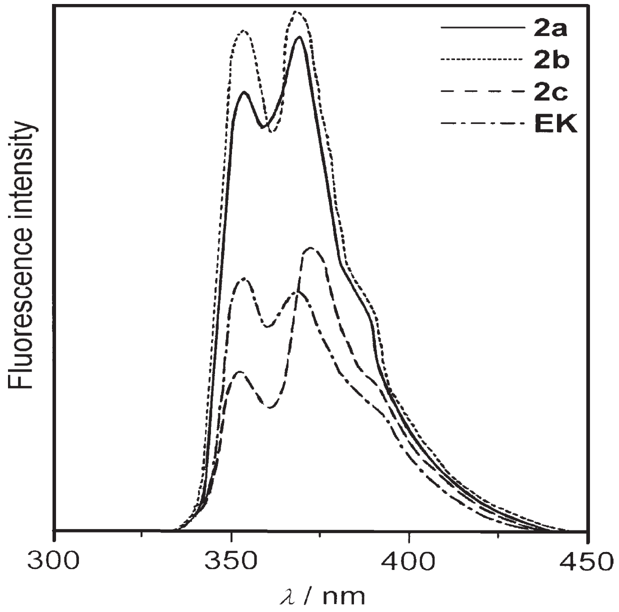


Figure 6. Fluorescence spectra of the dilute acetonitrile solutions (10^{-4} M) of compounds 2a-c and N-ethylcarbazole. λ_{ex} = 310 nm.

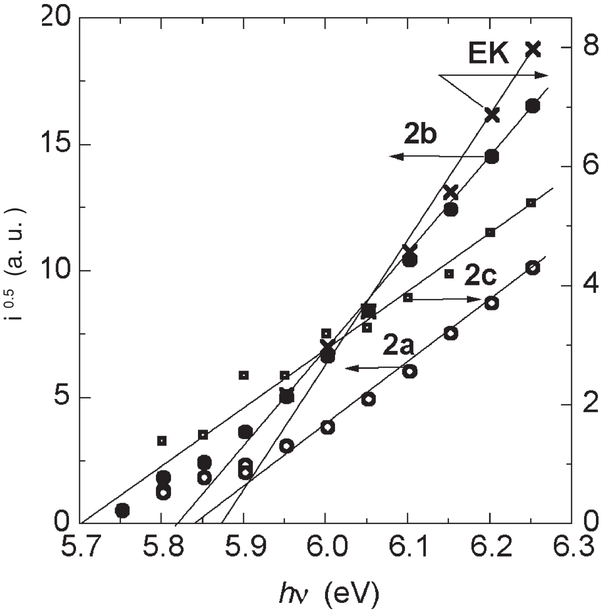


Figure 7. The electron photoemission in air spectra of the glassy layers of 2a, 2b and 2c. The spectrum of the crystalline layer of N-ethylcarbazole is given for the comparison.

The electron photoemission spectra of the glassy layers of the compounds investigated are presented in Fig. 7. The spectrum of the crystalline layer of EK is given for comparison. The I_p value for 2a is 5. 84 eV and is close to the 5. 8 eV value reported earlier for PVK. [23] The photoemission from crystalline EK is weak, but the I_p value approaches 5. 9 eV, also close to the value for the compound 2a. The photoemission from 2b is stronger, but the I_p value of 5. 82 eV is again very close to the I_p value for 2a. In the case of compound 2c the photoemission is much weaker than in the 2a case, and the I_p value may only be approximately estimated as not lower than 5. 7 eV. These results indicate, that the hole transport levels in carbazole, brominated carbazole and diphenylamine groups are energetically close. Accordingly, the presence of the different chromophores in such a molecule should not affect hole transport significantly. Results of the mobility measurements confirm this exception.

Conclusions

The amorphous carbazolyl-containing molecular glasses 2a-c have been synthesized by the reaction of glycidyl ethers 1a-c with resorcinol in the presence of triethylamine. The electrophotographic parameters of undoped films of the 2a-c and doped with DBBFP, which is regarded as one of the most effective sensitizers for carbazolyl-containing organic photoconductors, have been studied. Doping with DBBFP increases intrinsic photosensitivity of 2a-c in the UV region of the spectrum and induces photosensitivity in the visible region with a maximum at 425 nm. Despite the lower concentration of chromophores, the hole drift mobilities measured in these molecular glasses reaches 10^{-4} cm^2 V^{-1} s^{-1} at an electric field of 10^6 V cm^{-1} and thus exceeds the parameters of the well-known carbazolyl-containing polymers, PVK and PEPK. Another advantage of the synthesized molecular glasses 2a-c compared photoconductive oligomers and polymers is that they can be thoroughly purified by conventional methods of preparative organic chemistry.

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