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Synthesis and properties of glass-forming condensed aromatic amines with reactive functional groups

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Abstract

A series of new glass-forming condensed aromatic amines containing functional groups was synthesized by stepwise route and characterized by nuclear magnetic resonance, infrared and mass spectrometry. The thermal, optical and photoelectrical properties of the synthesized compounds are reported. The synthesized aromatic amines form glasses with the glass transition temperatures in the range of 73–104 °C as characterized by differential scanning calorimetry. The ionisation potentials of these compounds range from 5.25 to 5.36 eV as determined by electron photoemission method. The best charge transport properties were shown by di(4-(4-(3,6-di(*N*,*N*-diphenylamino)carbazole-9)-phenyloxy-2-hydroxypropylthio) phenyl)sulphide. Room temperature hole-drift mobilities in its 50% solid solutions in bisphenol Z polycarbonate established by the xerographic time-of-flight technique were found to be 2×10^{-6} cm^2 V^{-1} s^{-1} at electric field of 6.4×10^5 V cm^{-1}.

Keywords: Aromatic amine; Glass; Hole-drift mobility; Ionisation potential

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1. Introduction

Low molecular weight organic compounds that readily form stable glasses are called molecular glasses or amorphous molecular materials. They show excellent process ability, transparency, isotropic and homogeneous properties and receive growing attention both in terms of academic interest and technological applications. Aromatic amine-based molecular glasses belong to the class of hole-transporting materials which are known for various applications such as electrophotographic photoreceptors, organic light-emitting diodes, photovoltaic cells and photorefractive materials [1–3]. In multi-layer optoelectronic devices cross-linked charge transport or electroluminescent layers are preferable. Using cross-linkable materials upper layers can be prepared on the cross-linked under layer simple by casting or spin coating techniques. In addition, no diffusion between layers occurs when cross-linked materials are used. For the preparation of cross-linked layers organic electroactive materials with reactive functional groups are necessary.

In the present study, we report on the synthesis and investigation of the properties of glass-forming condensed aromatic amines with the functional hydroxyl and epoxy groups.

2. Experimental

2.1. Materials

The starting compounds: 9*H*-carbazole, diphenylamine, 4-iodoanisole, sodium hydride, ethanethiol, epichlorohydrin, 4,4′-thiobisbenzenethiol were purchased from "Aldrich" and used as received. All the required chemicals: 18-crown-6, potassium carbonate, copper powder, potassium iodide, potassium iodate, benzyltrimethylammonium chloride (BTMA), magnesium sulphate, triethylamine (TEA) were also purchased from "Aldrich". Organic solvents were purified and dried by standard methods [4]. Silica gel was used for column chromatography.

2.1.1. 9-(4-Methoxyphenyl) carbazole (*1*)

5.51 g (33 mmol) of carbazole, 10 g (43 mmol) of 4-iodoanisole, powdered potassium carbonate (36.43 g, 264 mmol), copper powder (8.38 g, 132 mmol) and 18-crown-6 (0.56 g, 2.1 mmol) were refluxed in 30 ml *o*-dichlorobenzene under nitrogen for 24 h. Then, copper and inorganic salts were filtered off. The solvent was removed by distillation. The product was crystallized from methanol, filtered and dried. Yield: 8.432 g (93.7%) of white crystals (mp: 154.1–155.4 °C). FW = 273 g mol^{-1}. IR (in KBr), ν (cm^{-1}): 3044, 3016 (CH$_{ar}$),

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2959, 2837 ($\mathrm{CH_{aliphatic}}$), 1593, 1514, 1479 ($\mathrm{C{=}C_{ar}}$), 1248 (C—O—C). MS (eV): $m/z = 274$ (M^+), 242, 166. ^{1}H NMR (300 MHz, $\mathrm{CDCl_3}$), δ (ppm): 4.04 (s, 3H, methoxy), 7.24–7.63 (m, 8H, $-\mathrm{CH_{ar}}$), 8.35–8.40 (m, 4H, $-\mathrm{CH_{ar}}$). ^{13}C NMR (75.4 MHz, $\mathrm{CDCl_3}$), δ (ppm): 55.95, 110.07, 115.44, 120.03, 120.68, 123.53, 126.25, 128.90, 130.65, 141.78, 159.26.

2.1.2. *3,6-Diiodo-9-(4-methoxyphenyl)-9H-carbazole (**2**)*

To a stirred mixture of 9-(4-methoxyphenyl) carbazole **1** (9.54 g, 34 mmol), potassium iodide (10.96 g, 66 mmol), acetic acid (150 ml), potassium iodate (16.05 g, 75 mmol) were added at 130 °C and the resulting mixture was stirred and heated for 1 h. After the reaction, most of the acetic acid was removed by rotary evaporation. The inorganic materials were dissolved in water. The organic product was filtered off, washed several times with *iso*-propanol and dried. Yield: 16.07 g (87.6%) of white powder **2**. FW = 525 g mol^{-1}. IR (in KBr), ν (cm^{-1}): 3055 ($\mathrm{CH_{ar}}$), 2995, 2933, 2836 ($\mathrm{CH_{aliphatic}}$), 1513, 1465 ($\mathrm{C{=}C_{ar}}$), 1230 (C—O—C). MS (eV): $m/z = 526$ (M^+), 271, 398, 107. ^{1}H NMR (100 MHz, $\mathrm{CDCl_3}$), δ (ppm): 3.94 (s, 3H, methoxy), 6.97–7.67 (m, 6H, $-\mathrm{CH_{ar}}$), 8.33–8.42 (m, 4H, $-\mathrm{CH_{ar}}$).

2.1.3. *3,6-Bis(diphenylamino)-9-(4-methoxyphenyl) carbazole (**3**)*

Six grams (11 mmol) of 3,6-diiodo-9-(4-methoxyphenyl)-9*H*-carbazole **2**, 3.72 g (22 mmol) of diphenylamine, powdered potassium carbonate (12.14 g, 88 mmol), copper powder (2.79 g, 44 mmol) and 18-crown-6 (0.6 g) were refluxed in 40 ml of *o*-dichlorobenzene under nitrogen for 24 h. Then, copper and inorganic salts were filtered off. The solvent was distilled. The product was crystallized from methanol, filtered and dried. Yield: 6.15 g (89%) of **3** (mp: 144 °C). FW = 607 g mol^{-1}. IR (in KBr), ν (cm^{-1}): 3035 ($\mathrm{CH_{ar}}$), 2932, 2836 ($\mathrm{CH_{aliphatic}}$), 1514, 1482 ($\mathrm{C{=}C_{ar}}$), 1225 (C—O—C). MS (eV): $m/z = 608$ (M^+), 439, 285. ^{1}H NMR (400 MHz, $\mathrm{CDCl_3}$), δ (ppm): 3.94 (s, 3H, methoxy), 6.95–7.40 (m, 26H, $-\mathrm{CH_{ar}}$), 7.42–7.59 (m, 2H, $-\mathrm{CH_{ar}}$), 7.90 (s, 2H, $-\mathrm{CH_{ar}}$). ^{13}C NMR (100 MHz, $\mathrm{CDCl_3}$), δ (ppm): 55.52, 110.72, 115.04, 118.69, 121.51, 122.61, 123.67, 125.89, 128.30, 128.98, 138.88, 140.24, 148.46, 154.87.

2.1.4. *3,6-Bis(diphenylamino)-9-(4-hydroxyphenyl) carbazole (**4**)*

A solution of sodium ethanethiolate was prepared by adding 4.43 ml (60 mmol) of ethanethiol to a suspension of 0.96 g (40 mmol) of sodium hydride (90%) in 70 ml of dry DMF at 0 °C under $\mathrm{N_2}$, and then by stirring for 15 min at room temperature. A solution of 2.5 g (4 mmol) of 3,6-bis(diphenylamino)-9-(4-metoxyphenyl)carbazole **3** in 15 ml DMF was added to the former solution and heated at 120 °C for 19 h. The reaction was stopped by carefully adding a few drops of water to the ice cooled solution. A further portion of water (150 ml) was added, followed by acidification with 2N HCl. The precipitated product was filtered off, washed with a large amount of hexane and purified by column chromatography (eluent:hexane/acetone, 3:1). Yield: 2.07 g (84.84%) of **4**. FW = 593 g mol^{-1}. IR (in KBr), ν (cm^{-1}): 3516 (OH), 3034 ($\mathrm{CH_{ar}}$), 1514, 1484 ($\mathrm{C{=}C_{ar}}$). MS (eV): $m/z = 594$ (M^+), 576, 257. ^{1}H NMR (300 MHz, $\mathrm{CDCl_3}$), δ (ppm): 5.50 (s, 1H, $-$OH), 7.00–7.40 (m, 26H, $-\mathrm{CH_{ar}}$), 7.42–7.59 (m, 2H, $-\mathrm{CH_{ar}}$), 7.90 (s, 2H, $-\mathrm{CH_{ar}}$). ^{13}C NMR (75.4 MHz, $\mathrm{CDCl_3}$), δ (ppm): 110.72, 116.55, 118.69, 121.54, 122.57, 123.67, 125.87, 128.53, 128.98, 138.88, 140.24, 148.46, 154.87.

2.1.5. *3,6-Bis(diphenylamino)-9-(4-[2,3-epoxypropyl]phenyloxy)carbazole (**5**)*

A mixture of 3,6-bis(diphenylamino)-9-(4-hydroxyphenyl)carbazole **4** (5 g, 8.43 mmol), epichlorohydrin (20 ml), BTMA (0.02 g) was heated at reflux for 1 h. Then, the excess of epychlorohydrin was removed at reduced pressure and the oil was dissolved in $\mathrm{CH_2Cl_2}$, washed several times with water and dried over magnesium sulphate. The solvent was evaporated under vacuum. The precipitated product was filtered off and purified by column chromatography (eluent:toluene/hexane, 4:1). Yield: 3.28 g (60%) of bright green powder **5**. FW = 649 g mol^{-1}. IR (in KBr), ν (cm^{-1}): 3034 ($\mathrm{CH_{ar}}$), 2922, 2853 ($\mathrm{CH_{aliphatic}}$), 1513, 1483 ($\mathrm{C{=}C_{ar}}$), 1225 (C—O—C). MS (eV): $m/z = 650$ (M^+), 481, 313, 149. ^{1}H NMR (300 MHz, $\mathrm{CDCl_3}$), δ (ppm): 2.79 (dd, 1H, in oxirane ring), 2.96 (dd, 1H, in oxirane ring), 3.41 (m, 1H, $-$OCH$-$), 3.99 (dd, 1H, in $-\mathrm{OCH_2}-$), 4.35 (dd, 1H, in $-\mathrm{OCH_2}-$), 6.85–7.47 (m, 30H, aromatic protons). ^{13}C NMR (75.4 MHz, $\mathrm{CDCl_3}$), δ (ppm): 44.86, 50.30, 69.33, 110.95, 116.00, 118.92, 121.77, 122.87, 123.97, 126.17, 128.98, 129.27, 139.10, 140.54, 148.76, 158.00.

2.1.6. *Di(4-(4-(3,6-di(N,N-diphenylamino)carbazole-9)-phenyloxy-2-hydroxypropylthio)phenyl)sulphide (**6**)*

One gram (1.54 mmol) of compound **5** and 0.19 g (0.77 mmol) of 4,4′-thiobisbenzenethiol were dissolved in 15 ml of ethylmethylketone. The reaction was catalyzed by adding of 0.5 ml of TEA at 80 °C. After 24 h, the reaction was terminated by precipitation into methanol. The precipitated product was filtered off and purified by column chromatography (eluent:hexane/acetone, 3:2). Yield: 1.5 g (63%) of **6**. IR (in KBr), ν (cm^{-1}): 3426 (OH), 3034 ($\mathrm{CH_{ar}}$), 2923, 2867 ($\mathrm{CH_{aliphatic}}$), 1512, 1482 ($\mathrm{C{=}C_{ar}}$). ^{1}H NMR (300 MHz, $\mathrm{CDCl_3}$), δ (ppm): 2.71 (m, 2H, $-$CH), 2.67 (s, 2H, $-$OH), 3.30 (m, 4H, $-\mathrm{SCH_2}$), 4.16 (s, 4H, $-\mathrm{OCH_2}$), 6.85–7.80 (m, 68H, $\mathrm{CH_{ar}}$). ^{13}C NMR (75.4 MHz, $\mathrm{CDCl_3}$), δ (ppm): 37.45, 68.58, 70.40, 110.73, 115.74, 118.74, 121.62, 122.71, 123.80, 125.97, 128.42, 128.48, 129.07, 130.34, 131.60, 133.94, 138.90, 140.42, 148.53, 157.53.

2.2. *Instrumentation*

Differential scanning calorimetry (DSC) measurements were carried out with a Mettler DSC-821e at 20 °C min^{-1} heating rate under $\mathrm{N_2}$ atmosphere. Thermogravimetric analysis (TGA) was performed on a Mettler TGA/SDTA 851e at a 20 °C min^{-1} heating rate, under nitrogen atmosphere. IR spectra were recorded using Perkin-Elmer FT-IR System. The spectra of solid compounds were performed in a form of KBr pellets. ^{1}H NMR and ^{13}C NMR spectra of deuterated chloroform and deuterated dimethylsulphoxide solutions were obtained on Fovies Trauspim (400 MHz (^{1}H), 100 MHz (^{13}C)), Varian Gemini (300 MHz (^{1}H), 75.4 MHz (^{13}C)), Varian Unity Inova (300 MHz (^{1}H), 75.4 MHz (^{13}C)) and JOEL JNM-FX 100 (100 MHz (^{1}H)) spectrometers. Electron impact mass spectra were obtained on a Waters 2Q 2000. UV–vis absorption spectra were recorded using Spectronic Genesys$^{\mathrm{TM}}$ 8 UV/VISIBLE spectrometer. Fluorescence emission and excitation spectra were recorded with Hitachi MPF-4 spectrometer.

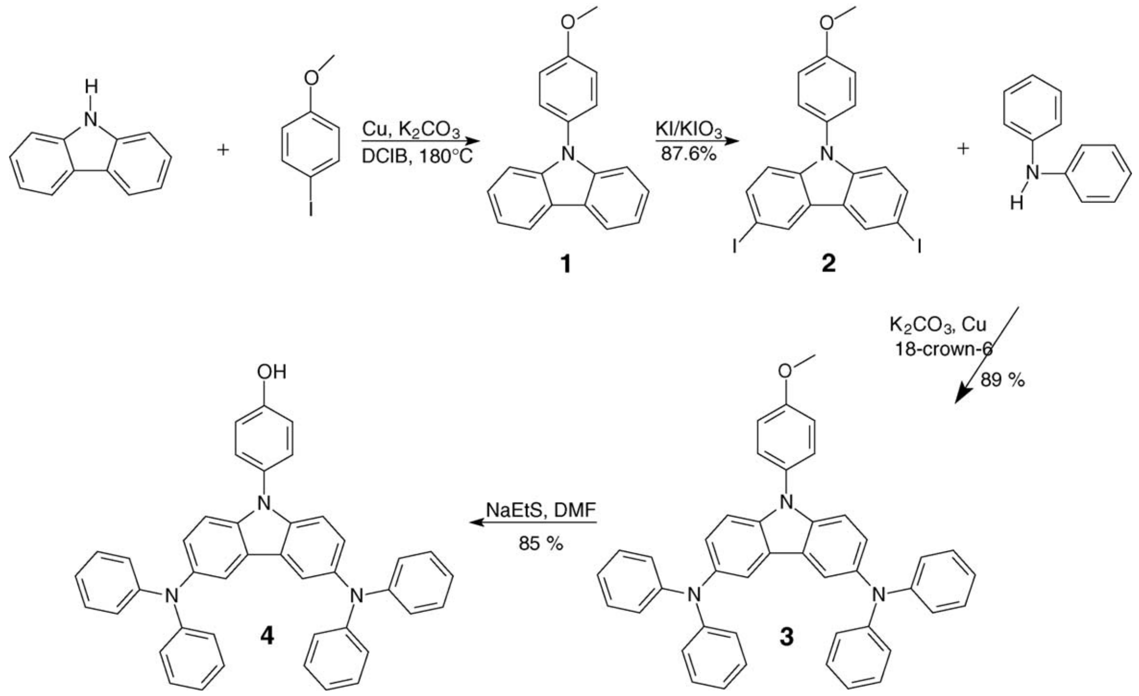
The ionisation potentials (I_p) of the films of the synthesized compounds were measured by electron photoemission in air method as described before [5,6]. The samples for the measurements were prepared by casting the solutions of the compounds on Al plates pre-coated with methylmethacrylate and methacrylic acid copolymer as adhesive layer.

Hole-drift mobilities were measured by a time-of-flight method in a xerographic regime [7,8]. Positive corona charging created electric field inside the TM layer. Charge carriers were generated at the layer surface by illumination with short UV pulses of xenon flash. The layer surface potential decrease as a result of pulse illumination was up to 3–5% of the initial potential before illumination. The transit time t_t was determined by the kink on the transient curve in double logarithmic scale. The drift mobility was calculated by the formula $\mu = d^2/U_0 t_\mathrm{t}$, where d is the layer thickness and U_0 is the surface potential at the moment of illumination. The samples for the measurements were prepared by casting the solutions of the compounds or solutions of the mixtures of these compounds with polycarbonate Z at mass proportion 1:1 in THF. The substrates were polyester films with Al layer.

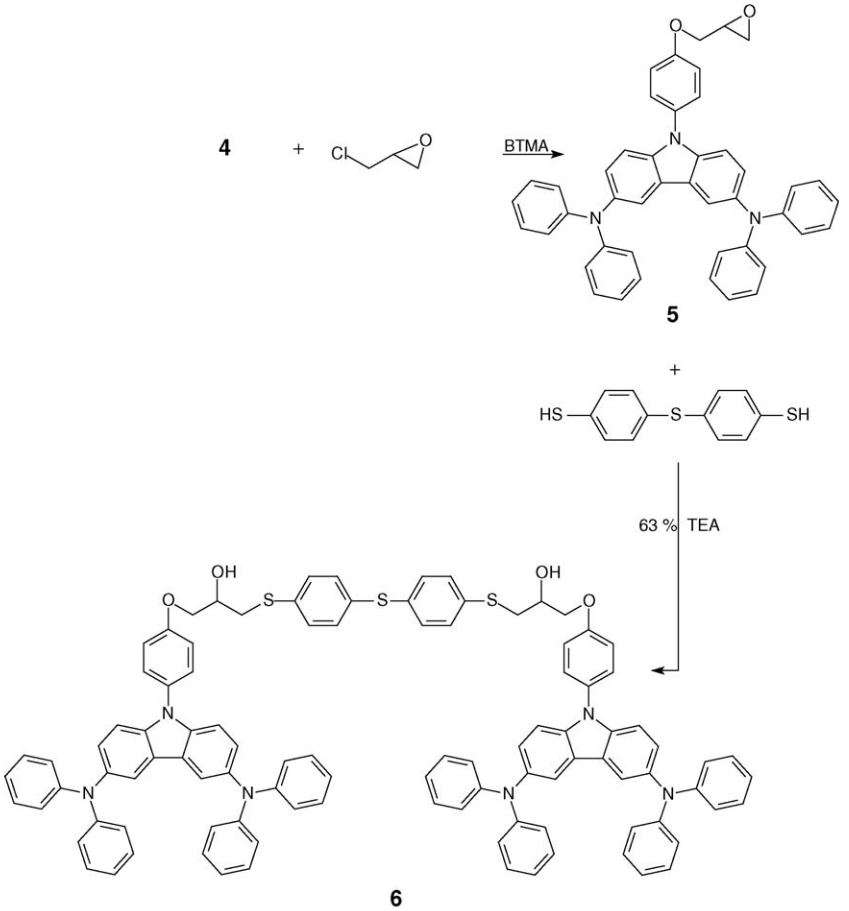
3. Results and discussion

3,6-Bis(diphenylamino)-9-(4-hydroxyphenyl)carbazole **4** was synthesized by three-step synthetic route as shown in Scheme 1. The first step was the iodination of 9-(4-methoxyphenyl)carbazole **1** using KI and $\mathrm{KIO_3}$. The second step was an Ullmann coupling of 3,6-diiodo-9-(4-methoxyphenyl)-9*H*-carbazole **2** with diphenylamine. The final step was demethylation of methoxy group of compound **3** with a solution of sodium ethanethiolate in dimethylformamide at ca. 120 °C. The product was purified by column chromatography.

Di(4-(4-(3,6-di(*N*,*N*-diphenylamino)carbazole-9)-phenyloxy-2-hydroxypropylthio) phenyl)sulphide **6** was synthesized as described in Scheme 2 by two-step procedure involving alkylation of compound **4** with an excess of epichlorohydrin



Scheme 1. Synthetic route to compounds **3** and **4**.



Scheme 2. Synthesis of compounds **5** and **6**.

Table 1
UV absorption (in THF solutions) and PL[a] emission (in THF solutions) maxima of the compounds **3–6** and PhCz[b]

Compounds	UV: λ_{max} [nm]	PL: λ_{max} [nm]
3	304, 358[c]	428, 447[c]
4	305, 358[c]	430, 449[c]
5	306, 360[c]	431, 450[c]
6	310, 365[c]	433, 452[c]
PhCz	240, 293, 340	300, 313

[a] Excitation wavelength 290 nm.
[b] 10^{-5} mol l^{-1}.
[c] Shoulder.

and the following addition reaction of 3,6-bis(diphenylamino)-9-(4-[2,3-epoxypropyl]phenyloxy)carbazole **5** and 4,4′-thiobisbenzenethiol in the presence of TEA as catalyst.

The structures of the newly synthesized compounds were confirmed by IR, ^{1}H NMR, ^{13}C NMR spectroscopy and mass spectrometry (see Section 2). They are soluble in common organic solvents such as chloroform, acetone and tetrahydrofuran.

The characteristic aromatic absorption at 3034–3059 cm^{-1} (C–H stretch), aliphatic absorption at 2836–2995 cm^{-1} (C–H stretches), at 1465–1516 cm^{-1} (C=C stretches) and absorption at 1310 cm^{-1} due to C–N stretching are clearly observed in the IR spectra of all aromatic amino groups containing compounds. Compounds **4** and **6** also have characteristic OH stretches at 3516 and 3426 cm^{-1}, respectively. The characteristic C–O–C bands at 1225 cm^{-1} are clearly observed in the IR spectra of **5**.

The signals in ^{1}H NMR spectra of the newly synthesized compounds **3–6** can be assigned to the characteristic aromatic and aliphatic hydrogen atoms. The proton signals at 3.94 and 5.50 ppm can be assigned to the protons methoxy group of compound **3** and hydroxy group of **4**, respectively. The signals at 6.80–8.00 ppm can be assigned to the aromatic protons.

The mass spectra of the newly synthesized compounds **3–5** show the corresponding molecular ion peaks.

The newly synthesized aromatic amines were also characterized by UV/VIS and PL spectrometry. The maxima of the UV absorption and fluorescence emission spectra of dilute THF solutions of compounds **3–6** are summarized in Table 1. For comparison, the maxima of UV absorption and PL emission of 9-*phenylcarbazole* (PhCz) are given in Table 1.

Aromatic amines **3–6** absorb light in the range of 220–350 nm (Fig. 1). The absorption spectra of compounds **3–6** are similar, due to the presence of the same chromophore. The highest values of molar extinction coefficient were observed for compound **6** having two chromophores in a molecule. The spectrum of compound **6** also exhibits the highest bathochromic shift with respect to the spectrum of compounds 9-phenylcarbazole.

The thermal properties of the synthesized aromatic amines were examined by DSC and TGA under a nitrogen atmosphere. The values of glass transition temperatures (T_g), melting points (T_m) and 5% weight loss temperatures (T_{ID}) are summarized in Table 2. All these materials demonstrate high thermal stability. They decompose in one step and the temperatures of 5% weight loss are higher than 300 °C, as confirmed by TGA with a heating rate of 20 °C min^{-1}.

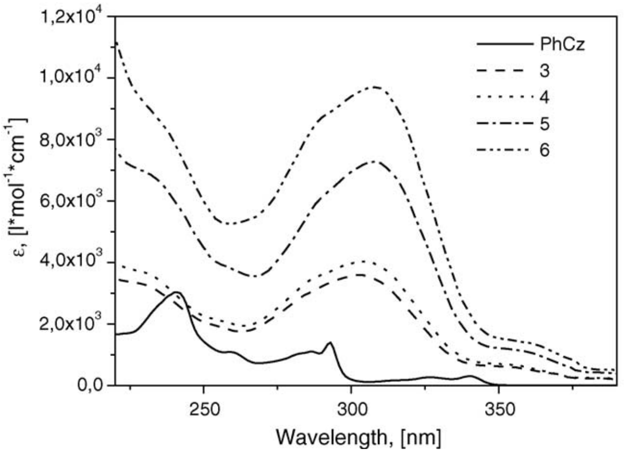


Fig. 1. UV absorption spectra of dilute THF solutions of compounds **3–6**.

Table 2
Thermal characteristics of the compounds **3–6**

Compounds	T_g [°C]	T_m [°C]	T_{ID}[a] [°C]
3	77.5	145	325
4	73	–[b]	384
5	78	–[b]	330
6	104	–[b]	

[a] T_{ID} is the temperature at which initial loss of mass (5%) was observed.
[b] Obtained as an amorphous material.

DSC thermograms of compound **3** are shown in Fig. 2.The first DSC heating run of the sample revealed melting peak with a maximum at 145 °C. Recooling revealed no crystallization peak and only glass transition was observed in the following DSC heating run with the glass transition temperature of 77.5 °C. Compounds **4–6** were isolated as amorphous materials and only glass transitions were observed in their DSC scans at 73 °C for **4**, at 78 °C for **5** and at 104 °C for **6**, and no peaks due to crystallization and melting appeared. Cooling down and the

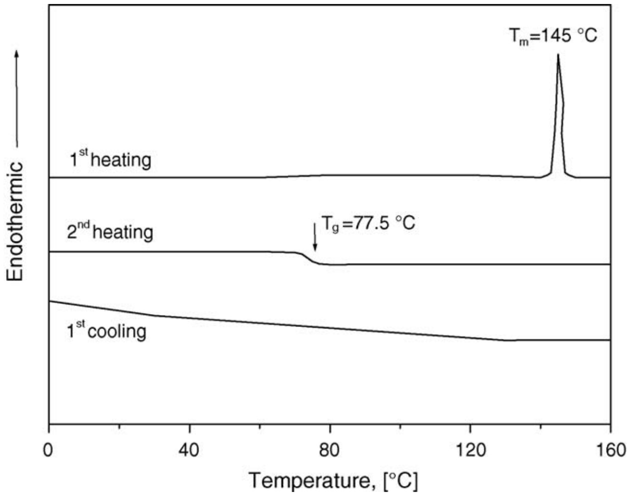


Fig. 2. DSC curves of **3**, at the heating/cooling rate of 20 °C min^{-1}, N_2 atmosphere.

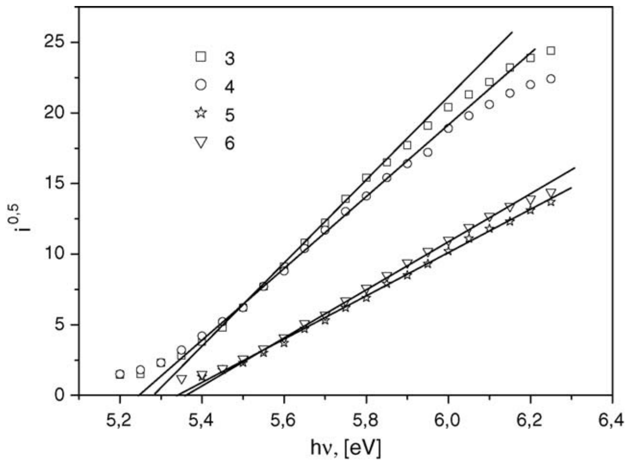


Fig. 3. The electron photoemission spectra in air of the films of compounds **3–6**.

following repeated heating revealed only the glass transition again.

An important characteristic of electronically active compounds used in optoelectronic devices is ionisation potential (I_p), which characterizes the electron releasing work under illumination. I_p values for the films of the synthesized compounds were established by electron photoemission technique from the dependencies of photocurrent (I) on the incident light quanta energy, which are named as electron photoemission spectra and plotted as $I^{0.5} = f(hv)$. The photoemission spectra of the amorphous films of the synthesized aromatic amines are shown in Fig. 3 and the values of ionisation potentials are summarized in Table 3.

The I_p values of the synthesized compounds are rather close. They are close to I_p values of charge generation materials widely used in electrographic photoreceptors such as titanyl phthalocyanines, perylene pigments and bisazo pigments (5.1–5.6 eV) [9]. This observation shows that the synthesized compounds can be used as charge transport materials in electrophotographic photoreceptors. Compounds **3–6** can apparently also be used as hole-transporting materials in organic light-emitting diodes. Their I_p values are rather close to that of indium–tin oxide (4.8 eV), which is used as anode in electroluminescent devices. The injection barrier of holes from the electrode into the layer of **3–6** would be 0.56–0.45 eV.

The xerographic time-of-flight technique was used to characterize hole-drift mobilities in the films of aromatic amines **5** and **6** molecularly doped in polymeric host PC-Z (50 wt.%). The room temperature hole-drift mobility of these materials shows linear dependencies on the square root of the electric field (Fig. 4). This observation is characteristic of the majority of non-crystalline organic semiconductors and is attributed to the effects of disorder on charge transport [10].

Table 3
Ionisation potentials of the compounds **3–6**

Compounds	I_p [eV]
3	5.29
4	5.25
5	5.35
6	5.36

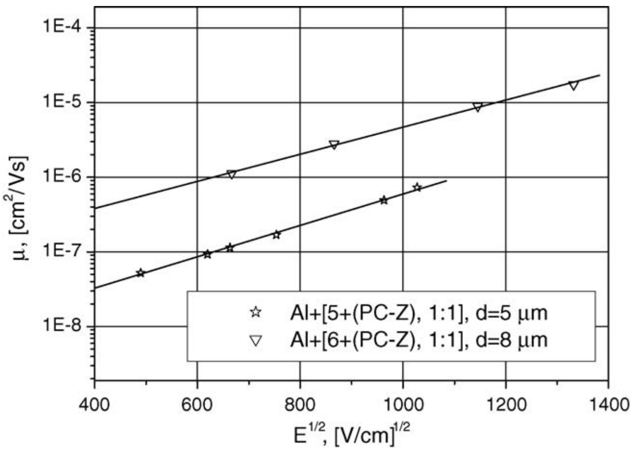


Fig. 4. Electric field dependency of the hole-drift mobilities (μ) in a charge transport layer of compounds **5** and **6** doped in polycarbonate (PC) (50 wt.%).

The values of hole-drift mobilities in 50% solid solutions of **5** and **6** in bisphenol Z polycarbonte were 2.2×10^{-7} and 2×10^{-6} cm^2 V^{-1} s^{-1}, respectively, at electric field of 6.4×10^5 V cm^{-1}.

4. Conclusions

New glass-forming condensed aromatic amines containing functional hydroxyl and epoxy groups were synthesized and characterized. The chemical structure of all the compounds reported was confirmed by ^{1}H NMR, ^{13}C NMR and IR spectroscopy. All these compounds were found to form glasses with glass transition temperatures in the range of 73–104 °C as established by differential scanning calorimetry. The ionisation potentials of these compounds range from 5.25 to 5.36 eV as determined by electron photoemission method. Time-of-flight experiment showed that hole-drift mobilities of 50% solid solutions of 3,6-bis(diphenylamino)-9-(4-[2,3-epoxypropyl]phenyloxy)carbazole and di(4-(4-(3,6-di(*N*,*N*-diphenylamino)carbazole-9)-phenyloxy-2-hydroxypropylthio) phenyl)sulphide in bisphenol Z polycarbonte were 2.2×10^{-7} and 2×10^{-6} cm^2 V^{-1} s^{-1}, respectively, at electric field of 6.4×10^5 V cm^{-1}.

Acknowledgements

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