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(Mechano)synthesis of azomethine- and terpyridine-linked diketopyrrolopyrrole-based polymers

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Abstract

Three efficient synthetic approaches towards new azomethine- and terpyridine-containing 2,5-dihydropyrrolo[3,4-c]pyrrole-1,4-dione (diketopyrrolopyrrole, DPP) based polymers, such as **P1** and **P2**, are reported. The first approach involves the Pd-catalyzed synthesis *via* two- or three-component Suzuki or Stille cross-coupling reaction in solution. The second approach involves Pd-catalyzed Suzuki cross-coupling reaction under ball-milling conditions. And, finally, the third approach involves Pd-free condensation reaction under ball-milling conditions. The newly obtained polymers exhibited absorbance around 700 nm and emission around 900 nm, and, thus, these polymers are considered to be NIR-fluorophores.

Keywords

diketopyrrolopyrrole-based polymers
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Supplementary materials

1. Introduction

2,5-Dihydropyrrolo[3,4-c]pyrrole-1,4-diones, diketopyrrolopyrroles, (DPPs) and DPP-based polymers have already found wide spread applications in various fields, including molecular electronics [1] and sensorics [2], optical studies [3], biological studies [4, 5] and others [6]. Therefore, new synthetic approaches to DPP derivatives are of high demand [1, 5, 7]. The most common approach to DPP-based polymers is Pd-catalyzed Stille, Heck or Sonogashira cross-coupling reactions in solution [5]. Less often Buchwald cross-coupling reaction [8] or Yamomoto polymerization [9] are used. The serious drawback of solution polymerization processes is low polymerization degrees (DPs) caused by fast precipitation [10], while the usage of solubilizing groups may influence the performance of the resulting polymer [11, 12].

In the last decade, the mechanosynthesis of polymers has attracted much attention due to such important advantages of mechanochemistry/solid state chemistry as environmental friendliness, the possibility of new chemical transformations, novel reactivity, etc. Many reports have already confirmed the suitability of mechanochemical approaches for the preparation of polymers of various geometry [13]. However, no examples were reported on mechanochemistry/solid state chemistry-based synthetic approaches towards DPP-based polymers. In this article we wish to report solvent-based and mechanochemical synthetic approaches to azomethine- and terpyridine-linked diketopyrrolopyrrole-based polymers.

2. Experimental

All reagents were purchased from commercial sources and used without further purification. Silica gel 60 (Kieselgel 60, 230–400 mesh) was used for the column chromatography. NMR spectra were recorded on a Bruker Avance-400 (or Bruker Avance-500) spectrometer, 298 K, digital resolution ± 0.01 ppm, using TMS as an internal standard. The ball mill was a Retsch PM 100 CM. UV-Vis spectra were recorded on a UV-2600 spectrophotometer (Shimadzu). Fluorescence spectra were recorded on a FS-5 spectrofluorometer (Edinburgh Instruments) and were corrected using Edinburgh correction curves. Mass spectra were recorded on a MicrOTOF-Q II mass spectrometer (Bruker Daltonics) with electrospray ionization. Elemental analyses were performed on a PE 2400 II CHN-analyzer (Perkin Elmer). GPC measurements were performed using a chromatograph Agilent 1200 with an aerosol light scattering detector (ELSD) (Agilent technologies, USA) and an Agilent Resipore column, 300x7.5 mm – 2 pieces in series. THF (stabilized with 0.0025% BHT) was used as an eluent with a flow rate of 1 ml/min. 2,6-Bis(6-(4-bromophenyl)-1,2,4-triazin-3-yl)pyridine [14] was synthesized as described in the literature.

2.1. 2,6-Bis(4-(4-bromophenyl)-6,7-dihydro-5*H*-cyclopenta[c]pyridin-1-yl)pyridine (2)

A mixture of 2,6-bis(6-(4-bromophenyl)-1,2,4-triazin-3-yl)pyridine (275 mg, 0.5 mmol) and 1-morpholinocyclopentene (0.8 ml, 5.0 mmol) was stirred at 150 °C under argon atmosphere for 2 h. Then an additional portion of 1-morpholinocyclopentene (0.4 mL, 2.5 mmol) was added and the resulting mixture was stirred under the same conditions for 1 h. The product was then purified by column chromatography (DCM, then mixture of DCM:ethyl acetate (1:9) as eluent, Rf 0.5). The analytical sample was obtained by recrystallization (MeCN). Yield 149 mg (0.24 mmol, 48%). **^{1}H NMR** ($CDCl_3$, δ, ppm): 2.08 (*m*, 4H, CH_2-6), 3.03 (*t*, 4H, ^3J 7.6 Hz, CH_2-7), 3.54 (*t*, 4H, ^3J 7.6 Hz, CH_2-5), 7.38–7.41 and 7.61–7.65 (both *m*, 4H, C_6H_4Br), 7.98 (*t*, 1H, ^3J 7.6 Hz, H-4(Py)), 8.17–8.20 (*m*, 2H, H-3,5(Py)), 8.54 (*s*, 2H, H-6′,6″(Py)). **ESI-MS**, *m/z*: found 622.05, calcd 622.05 $[M+H]^+$. Found, %: C 63.72, H 4.17, N 6.52. **$C_{33}H_{25}Br_2N_3$.** Calcd, %: C 63.58, H 4.04, N 6.64.

2.2. Monomer 6

A mixture of 3,6-Bis(5-bromo-2-thienyl)-2,5-bis(2-hexyldecyl)-2,5-dihydro-pyrrolo[3,4-c]pyrrole-1,4-dione **1** (41 mg, 0.045 mmol), aniline **4** (19.8 mg, 0.090 mmol) and $Pd(PPh_3)_4$ (5.2 mg, 10 mol %) was dissolved in dry 1,4-dioxane (10 mL). Then, a solution of K_2CO_3 (124.9 mg, 0.90 mmol) in water (4 mL) was added, and the resulting mixture was stirred in a round bottom pressure flask at 100 °C under argon atmosphere for 24 h. The resulting solution was washed with ethyl acetate, aqueous KOH, aqueous NH_4Cl and water. The organic phase was dried over anhydrous Na_2SO_4, filtered, and concentrated under reduced pressure. The obtained residue was purified by column chromatography using a mixture of ethyl acetate:hexane (2:3) as an eluent, R_f 0.35. The analytical sample was obtained by recrystallization (acetonitrile). M.p. 187–189 °C. Yield 30.0 mg (0.03 mmol, 71%). **^{1}H NMR** ($CDCl_3$, δ, ppm): 0.78–0.91 (*m*, 12H, CH_3), 1.17–1.38 (*m*, 48H, CH_2), 1.96–2.04 (*m*, 2H, $NH_2C\underline{H}$), 3.88 (*br. s*, 4H, NH_2), 4.06 (*d*, 4H, ^3J 7.2 Hz, $N\underline{H_2}CH$), 6.69–6.74 (*m*, 4H, $C_6H_4NH_2$), 7.30 (*d*, 2H, ^3J 4.2 Hz, thienyl), 7.47–7.52 (*m*, 4H, $C_6H_4NH_2$), 8.93 (*d*, 2H, ^3J 4.2 Hz, thienyl). **^{13}C NMR** ($CDCl_3$, δ, ppm): 14.1, 14.1, 22.6, 22.7, 26.4, 26.4, 29.3, 29.6, 29.7, 29.7, 30.1, 31.4, 31.8, 31.9, 37.9, 46.3, 107.7, 115.2, 115.5, 122.5, 127.3, 127.5, 136.9, 139.7, 147.3, 150.6, 161.8. **ESI-MS**, *m/z:* 931.60 $[M+H]^+$. Found, %: C 74.65, H 8.72, N 6.14. **$C_{58}H_{82}N_4O_2S_2$.** Calculated, %: C 74.79, H 8.87, N 6.02.

2.3. P1

A mixture of 2,6-bis(4-(4-bromophenyl)-6,7-dihydro-5*H*-cyclopenta[c]pyridin-1-yl)pyridine **2** (50 mg, 0.08 mmol), 2,5-bis(tributylstannyl)thiophene **3** (106.2 mg, 0.16 mmol) and dibromo-substituted DPP **1** (72.7 mg, 0.08 mmol) was dissolved in dry toluene (20 ml). The bis(triphenylphosphyne) palladium dichloride (5.6 mg, 0.008 mmol) and triphenylphosphine (4.2 mg, 0.016 mmol) were added, and the resulting mixture was stirred in a round bottom pressure flask at 100 °C under argon atmosphere for 50 h. The mixture was washed with aqueous solutions of ammonium chloride and dried with anhydrous sodium sulfate. The solvent was removed under low pressure. The residue was triturated with ethanol. The obtained precipitate was filtered off, washed with hexane, acetone and methanol, and dried. The residue was re-dissolved in chloroform and dried in vacuum to give a black-green solid. Yield 64 mg (0.05 mmol, 58%). **^{1}H NMR** ($CDCl_3$, δ, ppm): 0.79–0.95 (*m*, 25H), 1.10–1.42 (*m*, 84H), 1.90–2.00 (*m*, 2H), 2.05–2.14 (*m*, 4H), 3.00–3.14 (*m*, 4H), 3.42–3.49 (*m*, 4H), 3.97–4.07 (*m*, 3H), 7.38–7.43 (*m*, 2H), 7.56–7.65 (*m*, 4H), 7.73–7.81 (*m*, 2H), 7.99 (*t*, 1H, ^3J 7.6 Hz, H-4(Py)), 8.17–8.23 (*m*, 2H), 8.53–8.55 (*m*, 1H), 8.60–8.64 (*m*, 1H), 8.85–9.00 (*m*, 2H).

2.4. P2 (Method A)

A mixture of dibromo-substituted DPP **1** (100.0 mg, 0.11 mmol), aniline **4** (24.2 mg, 0.11 mmol) and terephthalic aldehyde **5** (14.8 mg, 0.11 mmol) was dissolved in a mixture of 1,4-dioxane (15 ml) and water (5 mL). Then bis(triphenylphosphyne) palladium dichloride (7.7 mg, 0.01 mmol), triphenylphosphine (5.8 mg, 0.02 mmol) and K_2CO_3 (152.4 mg, 1.10 mmol) were added, and the resulting mixture was stirred in a round bottom pressure flask at 100 °C under argon atmosphere for 36 h. The product was extracted by chloroform (2x30 mL); the organic layer was washed with aqueous solutions of KOH and ammonium chloride, dried over anhydrous sodium sulfate and filtered. The resulting solution was evaporated to dryness under low pressure, and the obtained residue was triturated with ethanol. The obtained precipitate was filtered off, washed with hexane, acetone and methanol, and dried. The residue was re-dissolved in chloroform and evaporated in vacuum to give a dark-green solid. Yield 28.0 mg (0.027 mmol, 25%). **^{1}H NMR** ($CDCl_3$, δ, ppm): 0.79–0.91 (*m*, 15H), 1.11–1.31 (*m*, 54H), 1.48–1.73 (*m*, 18H), 1.95–2.05 (*m*, 2H), 4.01–4.12 (*m*, 3H), 6.68–6.73 (*m*, 1H), 7.28–7.37 (*m*, 10H), 7.43–7.51 (*m*, 11H), 7.51–7.58 (*m*, 5H), 7.63–7.76 (*m*, 11H), 7.96–8.07 (*m*, 2H), 7.50–7.60 (*m*, 1H), 8.90–9.00 (*m*, 1H).

2.5. P_2 (Method B)

A mixture of dibromo-substituted DPP **1** (44.0 mg, 0.05 mmol), aniline **4** (10.6 mg, 0.05 mmol) and terephthalic aldehyde **5** (6.5 mg, 0.05 mmol), Pd(OAc)2 (1.1 mg, 0.005 mmol) and potassium carbonate (134.1 mg, 0.97 mmol) was grinded in a stainless steel 25 mL jar with 4 stainless steel 10-mm diameter balls at 500 rpm speed for 4 h at ambient temperature. The sample was treated by chloroform, and the organic solution was washed with an aqueous solutions of ammonium chloride and dried with anhydrous sodium sulfate. The solvent was removed under low pressure. The residue was triturated with ethanol. The obtained precipitate was filtered off, washed with hexane, acetone and methanol, and dried. The residue was dissolved in chloroform, evaporated to dryness, treated with ethanol, filtered and dried under vacuum to give a dark-green solid. Yield 30 mg (0.029 mmol, 60%).

2.6. P2 (Method C)

A mixture of diamino-substituted DPP **6** (93.0 mg, 0.10 mmol), terephthalic aldehyde **5** (13.4 mg, 0.10 mmol), *p*-toluenesulfonic acid (1.0 mg, 0.005 mmol) and $CaCl_2$ (110.8 mg, 1.0 mmol) was grinded in a 25 mL stainless steel jar with 4 stainless steel 10-mm diameter balls at 500 rpm for 4 h at ambient temperature. The sample was treated by dichloromethane, and the organic solution was washed with an aqueous solutions of saturated ammonium chloride and dried with anhydrous sodium sulfate. The solvent was removed under low pressure. The residue was triturated with ethanol. The obtained precipitate was filtered off, washed with hexane, acetone and methanol, and dried. The residue was re-dissolved in dichloromethane, evaporated to dryness, suspended in ethanol, filtered and dried under vacuum to give a dark-green solid. Yield 88 mg (0.085 mmol, 85%).

2.7. P2 (Method D)

Trifluoroacetic acid (3 drops) was used instead of p-TSA.

3. Results and Discussion

As a first step, polymer **P1** was prepared by using three-component Stille cross-coupling. Thus, equimolar amounts of dibromo-substituted DPP **1** and 2,6-bis(4-(4-bromophenyl)-6,7-dihydro-5*H*-cyclopenta[c]pyridin-1-yl)pyridine **2** and 2,5-bis(tributylstannyl)thiophene **3** (2 mol. Eq) were heated in dry toluene at 100 °C in the presence of $Pd(PPh_3)_2Cl_2$ (10 mol.%) and PPh_3 (20 mol.%) for 50 h to afford **P1** as a greenish solid in 58% yield. In case of polymer **P2** a three component Suzuki cross-coupling was used by means of heating equimolar amounts DPP **1**, 4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)aniline **4** and terephthalic aldehyde **5** in the presence of $Pd(OAc)_2$ (10 mol.%), PPh_3 (20 mol.%) and K_2CO_3 (10 mol. Eq) at 100 °C in 1,4-dioxane:water (4:1) solution for 36 h to afford **P2** as a greenish solid in as low as 25% yield. It is worth to mention that a partial precipitation during the reaction took place. To overcame the low-solubility limitations, we carried out the same reaction in a solvent-free manner under ball-milling conditions. To do that the equimolar amounts of compounds were reacted in the presence of $Pd(OAc)_2$ (10 mol.%) and K_2CO_3 (20 mol. Eq) at 500rpm for 4h to afford the polymer **P2** in 60% yield. It is worth mentioning that, in some cases, the polymers obtained *via* Pd-catalyzed polymerizations might be contaminated with some residual Pd, which might have a negative [15] or positive influence on the performance of the obtained polymer [16]. Moreover, owing to a wide spread use of DPP-polymers for biological applications, their contamination with traces of cytotoxic Pd is unacceptable. Therefore, as a last step, we developed a Pd-free synthesis of polymer **P2**. To do that, we prepared an aniline-appended DPP **6** by using Suzuki cross-coupling reaction between **1** and **4**. Next, compound **6** was reacted with terephthalic aldehyde in the presence of *p*-toluenesulphonic acid (p-TSA) (5 mol.%) and excess of $CaCl_2$ as dehydrating agent to afford the target polymer **P2** in as high as 85% yield. It should be noted that trifluoroacetic acid (TFA) can be also used to catalyze the reaction, while the presence of $CaCl_2$ as a dehydrating agent and the milling speed of 500 rpm are critical for achieving good conversion.

The obtained polymers were fully characterized by means of ^1H NMR- and UV-spectroscopy, as well as gel-penetration chromatography (GPC). Thus, in ^1H NMR signals of protons of DPP moiety as well as terpyridine (for **P1**) and imine (for **P2**) were observed (Figures S3–S4, SI). In UV-spectra strong absorption bands were observed at 710 nm (for **P1**) and 660 nm (for **P2**) (Figure 1A, 1C), and sharp emission peaks were observed in NIR area, such as 874 nm (for **P1**) and 872 nm (for **P2**) (Figure 1B, 1D). Based on the data of GPC, the molecular weights of the obtained polymers **P1-P2** vary from 4 to 20kDa (Table 1). It is noteworthy that during the GPC experiments a partial precipitation of **P2** from the THF solution was observed. We can, therefore, suppose that the insoluble fraction consists of a highest molecular weight fraction. The lower solubility of the polymer **P2** is, probably, due to the lower content of solubilizing moieties per monomer unit, compared to the polymer **P1**. Based on all above mentioned, the mechanochemical polymerization could be a good alternative to the conventional solution-based methods, especially in case of polymers with lower content of solubilizing groups.

Table 1 Experimental data for the polymers **P1–P2.**

No.	Absorption, nm	Emission, nm	Molecular weight, kDa		PDI
			M_W	M_n	
P1	715	874	19836	11052	1.79
P2	610(sh.), 675	872	3709[a]	1065	2.84

[a] A partial precipitation of polymer **P2** took place during GPC-experiments.

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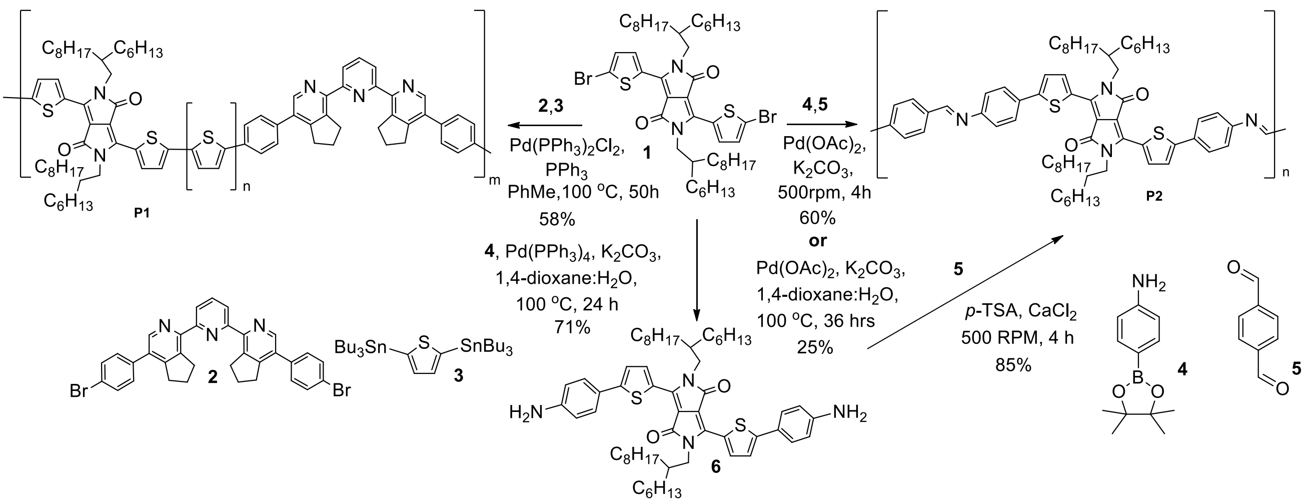

Scheme 1 Synthesis of DPP-based polymers **P1**, **P2**.

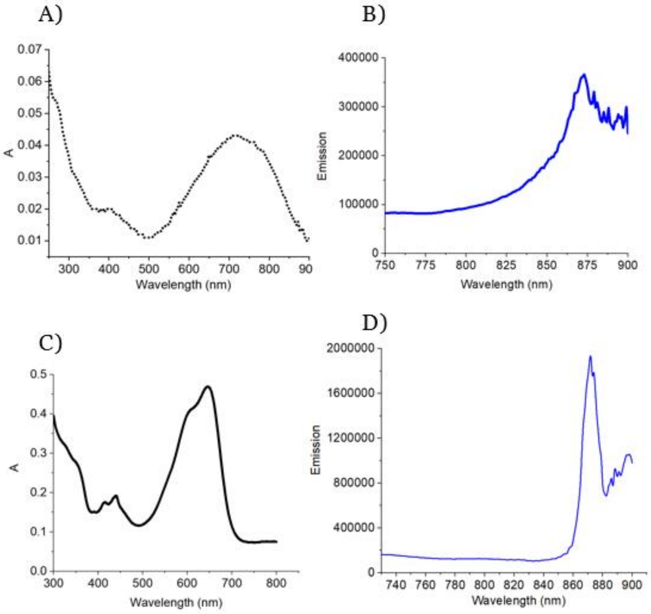

Figure 1 Photophysical data for polymers **P1–P2** in THF solution: absorption spectrum of **P1** (A); emission spectrum of **P1** (B); absorption spectrum of **P2** (C); emission spectrum of **P2** (D).

4. Limitations

The limitations of solvent-based processes are the long reaction time and the partial precipitation of resulting polymers during the reaction. To overcome these limitations, we carried out the reaction under ball-milling conditions with much shorter reaction time. In some cases, the polymers might contain some residual Pd, which, however, might have a negative [15] or positive effect on polymer performance [16]. To avoid these limitations, we carried out a Pd-free synthesis of polymer **P2** under ball milling conditions. In addition, a partial precipitation of polymer **P2** occurred during the GPC experiments, so the molecular weight determination might not be quite accurate.

5. Conclusions

In summary, by using solution-based three-component Stille or Suzuki cross-coupling, mechanochemical Suzuki cross-coupling or the Pd-free mechanochemical polycondensation reaction, azomethine- and terpyridine-linked diketopyrrolopyrrole-based polymers were prepared in up to 85% yields. Among all the methods used, the mechanosynthesis-based approach seemed to be the most convenient method for the preparation of low-soluble polymer **P2**, as it provided the highest conversion for a shortest reaction time. According to the results of the photophysical studies, both polymers exhibit strong absorption in 675–710 nm region, and their emission was observed at 872–875 nm. So, both polymers can be considered promising for the NIR-dyes for biovisualization.

• Supplementary materials

This manuscript contains supplementary materials, which are available on the corresponding online page.

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• Conflict of interest

The authors declare no conflict of interest.

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