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Synthesis and characterization of new polymeric phthalocyanines substituted with pyridine through methyleneoxy bridges by microwave irradiation

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Abstract

A tetranitrile monomer was synthesized by nucleophilic aromatic substitution of pyridine-2,6-diyldimethanol onto 4-nitrophthalonitrile. A metal-free phthalocyanine polymer was prepared by the reaction of a tetranitrile monomer with 4,4′-[pyridine-2,6-diylbis(methyleneoxy)]-diphthalonitrile in DMAE. Ni(II), Co(II), Cu(I)-phthalocyanine polymers were prepared by reaction of the tetranitrile compound with the chlorides of Ni(II), Co(II) and Cu(I) in DMAE. The Zn(II)-phthalocyanine polymer was prepared by reaction of the tetranitrile compound with the acetates of Zn(II) in DMAE. The new compounds were characterized by a combination of IR, ^{1}H NMR, ^{13}C NMR, UV–vis, elemental analysis and MS spectral data.
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Keywords: Polymeric phthalocyanine; Pyridine-2,6-diyldimethanol; Bisphthalonitrile; Tetranitrile; Metal-free phthalocyanine

1. Introduction

In addition to their industrial importance as pigments, metal phthalocyanines have been extensively studied because of their interesting conductivity, catalytic, photovoltaic, and electrochromic properties [1]. The physical and chemical properties of soluble phthalocyanines (Pc) have recently attracted attention from materials chemists for their potential use in semiconducting materials, nonlinear optics, and other optical devices [2].

Compared to low molecular weight phthalocyanines, relatively few reports describe the synthesis and properties of polymeric phthalocyanines. For the polycyclotetramerization, bifunctional monomers based on tetracarbonitriles for polymers [3–8], various oxy-, arylenedioxy- and alkylenedioxy-bridged diphthalonitriles for polymers [9–13] and other nitriles [14–16] have been employed, mainly in the presence of metal salts or metals. Polymeric phthalocyanines can only be utilized in some fields because of their insolubility in water and common solvents [17].

High-speed synthesis with microwave irradiation has attracted a considerable amount of attention in recent years [18]. Microwave irradiation is an electromagnetic irradiation in the frequency range of 0.3–300 GHz. The energy of the microwave photon in this frequency region (0.0016 eV) is too low to break the chemical bonds and is also lower than the energy of Brownian motion. It is therefore clear that microwave cannot induce chemical reactions [19–21]. Microwave assisted synthesis of phthalocyanines is novel [22–27].

We have previously synthesized phthalocyanines containing dithiadiaza [28] and tetraaza [29] macrocyclic moieties and pyridyl groups [30] by microwave irradiation. In this paper, we describe the synthesis and characterization of metal-free (**4**) and metallophthalocyanine polymers (**5–8**) by microwave irradiation containing pyridine-2,6-diyldimethanol moieties.

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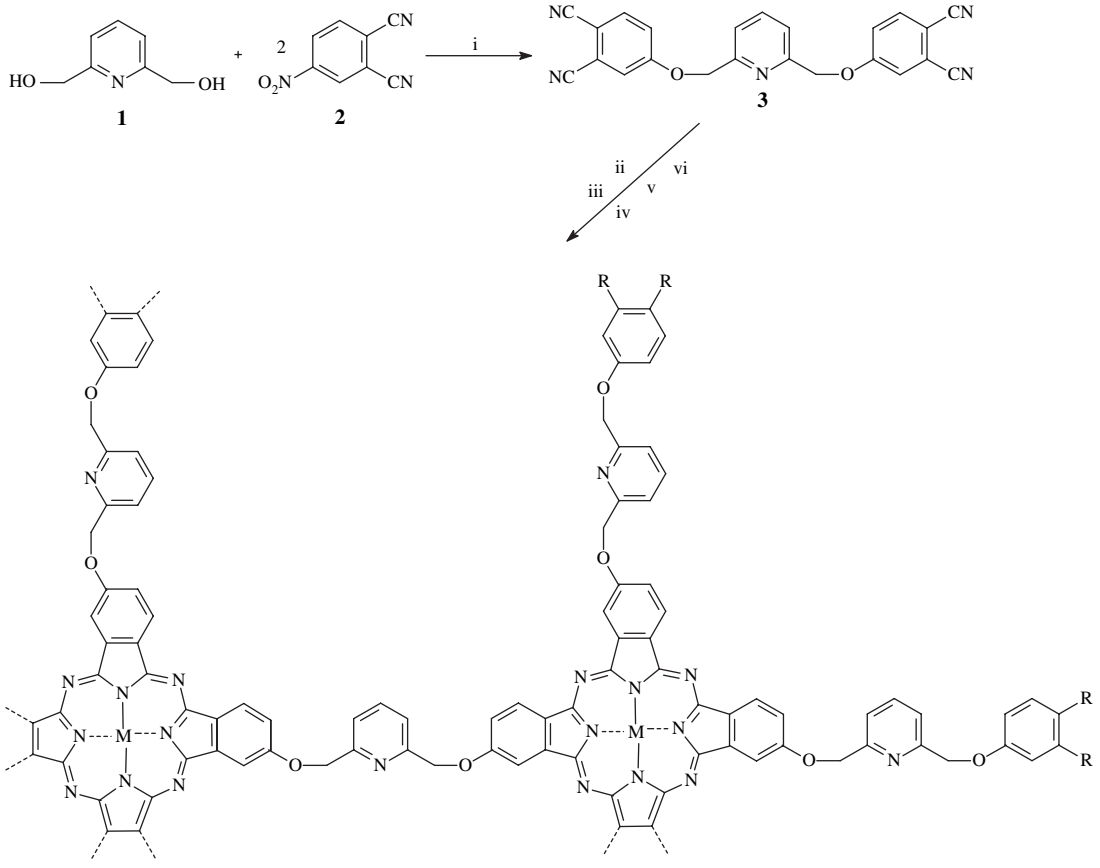
2. Results and discussion

Metal-free and metallophthalocyanine polymers were synthesized in two steps (Scheme 1). In the first step, the base-catalyzed nucleophilic aromatic nitro displacement [31,32] of 4-nitrophthalonitrile [33] **2** with pyridine-2,6-diyldimethanol [34] **1** afforded the tetranitrile monomer **3**. In the second step, metal-free and metallophthalocyanine polymers were synthesized. The metal-free phthalocyanine **4** was synthesized by microwave irradiation of the corresponding tetracyano compound **3** in 2-(dimethylamino)ethanol for 13 min. The metallophthalocyanines **5**–**8** were obtained from tetracyano derivative **3** and corresponding anhydrous metal salts $NiCl_2$, $Zn(CH_3COO)_2$, $CoCl_2$ and CuCl, respectively, by microwave irradiation in 2-(dimethylamino)ethanol for 12 min.

The molecular weights of the polymers could not be determined using traditional methods because of their poor solubility in organic solvents, but also by comparison of the IR absorption bands of the end groups with those of the bridging groups [9,35–39].

In the IR spectrum of **3** the disappearance of NO_2 and OH stretches, along with the appearance of new bands at 2232, 1597 and 1253 cm^{-1} arising from C≡N, C═N and Ar-O–C groups, respectively, are in agreement with the proposed structure. In the ^1H NMR spectrum of **3**, OH group of compound **1** disappeared as expected. ^1H NMR spectrum of **3** showed new signals at $\delta = 7.95$ (d, 2H, Ar-H), 7.73 (d, 2H, Ar-H), 7.69 (s, 2H, Ar-H), 7.57 (t, 1H, Ar-H), 7.38 (d, 2H, Ar-H), 5.36 (s, 4H, $-CH_2-O$). ^{13}C NMR spectrum of **3** indicated the presence of nitrile carbon atoms in **3** at



R: C≡N

R:

Compound	4	5	6	7	8
M	2H	Ni	Zn	Co	Cu

i: dry DMF, dry K_2CO_3, N_2, 50 °C
ii: Microwave, 350 W, 13 min, DMAE, 175 °C
iii: Microwave, 350 W, 12 min, DMAE, 175 °C, anhydrous $NiCl_2$
iv: Microwave, 350 W, 12 min, DMAE, 175 °C, anhydrous $Zn(CH_3COO)_2$
v: Microwave, 350 W, 12 min, DMAE, 175 °C, anhydrous $CoCl_2$
vi: Microwave, 350 W, 12 min, DMAE, 175 °C, anhydrous CuCl

Scheme 1.

$\delta = 115.14$ ppm. Elemental analysis and EI mass spectral data were satisfactory: 392 $[M + 1]^+$, 414 $[M + Na]^+$.

In the IR spectrum of **4** characteristic peaks for phthalocyanines were observed. The peak at 3276 cm^{-1} is the characteristic metal-free phthalocyanine N–H stretching band. Also, 1596 cm^{-1} (pyridine C=N) and 1253 cm^{-1} (Ar–O–C) bands were present in the spectrum. Elemental analysis was satisfactory.

The IR spectra of the metallophthalocyanine polymers **5**–**8** were very similar, except the metal-free phthalocyanine polymer **4** which showed an N–H stretching band at 3276 and 1089 cm^{-1} due to the inner core [40,41]. These bands disappear in spectra of the metallophthalocyanine polymers. These bands are especially beneficial for the characterization of metal-free phthalocyanine polymers, as there is little frequency dependence on ring substitution and they are not overlapped by strong bisphthalonitrile monomer absorptions [4]. The end groups of the metal-free phthalocyanine polymer were cyano groups (2232 cm^{-1}) while the end groups of the metallophthalocyanine polymers were imido groups ($\sim$1779–1703 cm^{-1}). We used a domestic oven synthesis of compounds **4**–**8** including 350 W, 175 °C as conditions.

In general, phthalocyanines show typical electronic spectra with two strong absorption regions, one in the UV region at about 300–500 nm related to the B band and the other in the visible region at 600–700 nm related to the Q band [42]. The split Q bands in **4**, which are characteristic for metal-free phthalocyanines were observed at $\lambda_{max} = 707$ and 683 nm. These Q band absorptions show the monomeric species with D_{2h} symmetry due to the phthalocyanine ring related to the fully conjugated 18π electron system [43–45]. The presence of strong absorption bands in **4** in the near UV region at $\lambda_{max} = 617$, 325 and 243 nm also shows Soret region B bands which have been ascribed to the deeper π–π* levels of LUMO transitions.

The UV–vis absorption spectra of metallophthalocyanines **5**–**8** in pyridine show intense Q absorption at $\lambda_{max} = 689$, 692, 689 and 688 nm, with a weaker absorptions at 623, 632, 623 and 618 nm, respectively. The single Q bands in metallo derivatives **5**–**8** are characteristic. This result is typical of metal complexes of substituted and unsubstituted metallophthalocyanines with D_{4h} symmetry [46]. B band absorptions of compounds **5**–**8** were observed at $\lambda_{max} =$ (392, 305 and 235), (368 and 313), (365 and 252), (348 and 275) nm as expected, respectively (Fig. 1).

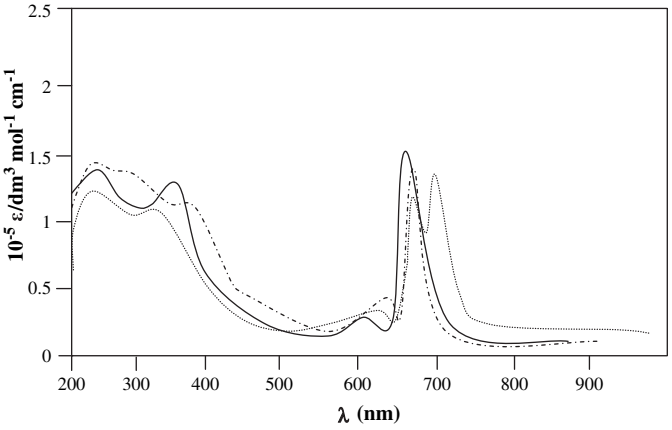


Fig. 1. UV–vis spectra of compounds **4** (···), **5** (–·–·–) and **7** (—) in pyridine.

3. Experimental

3.1. General methods

4-Nitrophthalonitrile [33] **2** and pyridine-2,6-diyldimethanol [34] **1** were prepared according to the literatures. All reagents and solvents were of reagent grade quality and were obtained from commercial suppliers. All solvents were dried and purified as described by Perrin and Armarego [47]. The IR spectra were recorded on a Perkin–Elmer 1600 FT-IR spectrophotometer, using KBr pellets or NaCl disc. ^{1}H and ^{13}C NMR spectra were recorded on a Varian Mercury 200 MHz spectrometer in $CDCl_3$, DMSO, and chemical shifts were reported (δ) relative to Me_4Si as an internal standard. Mass spectra were measured on a Micromass Quatro LC/ULTIMA LC–MS/MS spectrometer. Elemental analyses were determined by a LECO Elemental Analyser (CHNS O932) and Unicam 929 AA spectrophotometer. Melting points were measured on an electrothermal apparatus and are uncorrected. Optical spectra in the UV–vis region were recorded with a Unicam UV2-100 spectrophotometer, using 1 cm path length cuvettes at room temperature. Domestic oven was used for synthesis of all phthalocyanines.

3.2. Syntheses

3.2.1. 4,4′-[Pyridine-2,6-diylbis(methyleneoxy)]-diphthalonitrile (3)

Pyridine-2,6-diyldimethanol **1** (2 g, 14.37 mmol) was dissolved in dry DMF (80 ml) under N_2 and 4-nitrophthalonitrile **2** (4.97 g, 28.74 mmol) was added to the solution. After stirring for 10 min, finely ground anhydrous K_2CO_3 (5.94 g, 43.11 mmol) was added portionwise within 2 h with efficient stirring. The reaction mixture was stirred under N_2 at 50 °C for 5 d. After cooling, the reaction mixture was poured into 50 ml cold HCl (5 wt.%) solution to yield a crude product. This precipitate was stirred at room temperature for 24 h. At the end of this time, the precipitate was isolated by filtration and was first washed with distilled water, until the filtrate was neutral, and then Et_2O before drying in vacuo over P_2O_5. The crude product was purified by column chromatography on silica gel [chloroform/petroleum ether/methanol (7:2:1)]. Yield: 3.09 g (55%), mp: 228–230 °C. Anal. Calcd for $C_{23}H_{13}N_5O_2$: C, 70.58; H, 3.34; N, 17.89%. Found: C, 70.92; H, 3.02; N, 17.44. IR (KBr tablet) ν_{max}/cm^{-1}: 3075 (Ar-H), 2923–2846 (Aliph. C–H), 2232 (C≡N), 1597 (C=N), 1485, 1310, 1253, 1093, 850, 527. ^{1}H NMR ($CDCl_3$) (δ ppm): 7.95 (d, 2H, Ar-H), 7.73 (d, 2H, Ar-H), 7.69 (s, 2H, Ar-H), 7.57 (t, 1H, Ar-H), 7.38 (d, 2H, Ar-H), 5.36 (s, 4H, $-CH_2-O$). ^{13}C NMR ($CDCl_3$) (δ ppm): 161.86, 159.78, 138.18, 135.69, 124.61, 120.42, 120.30, 116.95,

116.02, 115.14, 110.46, 71.34. MS (EI) (*m/z*): 392 $[M+1]^+$, 414 $[M+Na]^+$.

*3.2.2. Metal-free polymeric phthalocyanine (**4**)*

A mixture of 4,4′-[pyridine-2,6-diylbis(methyleneoxy)]-diphthalonitrile **3** (0.5 g, 1.27 mmol) and 2-(dimethylamino) ethanol (DMAE) (4.5 ml) was irradiated in a microwave oven at 175 °C, 350 W for 13 min. After cooling to room temperature the reaction mixture was refluxed with ethanol to precipitate the product which was filtered off. The green solid product was washed with hot ethanol (45 ml) and dried in vacuo. This product is soluble in H_2SO_4 and hot pyridine. Yield: 517 mg (51%), mp > 300 °C. Elemental analyses (for CN end groups) $(C_{92}H_{54}N_{20}O_8)n$ $(1567.57)_n$ calcd: C, 70.49; H, 3.47; N, 17.87%; found: C, 70.04; H, 3.76; N, 18.22. IR (KBr tablet) ν_{max}/cm^{-1}: 3276 (N–H), 3076 (Ar-H), 2912–2846 (Aliph. C–H), 1596 (C=N), 1561, 1485, 1310, 1253 (Ar-O–C), 1089, 847, 526. UV–vis (pyridine) λ_{max}/nm: $[(10^{-5}\varepsilon\ dm^3\ mol^{-1}\ cm^{-1})]$: 243 (5.10), 325 (5.07), 618 (4.56), 683 (5.11), 707 (5.14).

*3.2.3. Ni(II)-containing polymer (**5**)*

A mixture of 4,4′-[pyridine-2,6-diylbis(methyleneoxy)]-diphthalonitrile **3** (0.5 g, 1.27 mmol), anhydrous $NiCl_2$ (82.2 mg, 0.63 mmol) and 2-(dimethylamino)ethanol (DMAE) (3 ml) was irradiated in a microwave oven at 175 °C, 350 W for 12 min. After cooling to room temperature the reaction mixture was refluxed with ethanol to precipitate the product which was filtered off. The green solid product was washed with hot ethanol (40 ml) and dried in vacuo. This product is soluble in H_2SO_4 and hot pyridine. Yield: 580 mg (63%), mp > 300 °C. Elemental analyses (for imide end groups) $(C_{92}H_{56}N_{16}O_{16}Ni)n$ $(1700.24)_n$ calcd: C, 64.99; H, 3.31; N, 13.18; Ni, 3.45%; found: C, 65.36; H, 2.98; N, 13.44; Ni, 3.62. IR (KBr tablet) ν_{max}/cm^{-1}: 3419 (imide N–H), 3081 (Ar–H), 2917–2851 (Aliph. C–H), 1772 (sym. C=O), 1720 (asym. C=O), 1605 (C=N), 1459, 1319, 1263 (Ar-O–C), 1083, 851, 534. UV–vis (pyridine): λ_{max}/nm: $[(10^{-5}\varepsilon\ dm^3\ mol^{-1}\ cm^{-1})]$: 235 (5.16), 305 (5.14), 392 (5.10), 623 (4.63), 689 (5.15).

*3.2.4. Zn(II)-containing polymer (**6**)*

A mixture of 4,4′-[pyridine-2,6-diylbis(methyleneoxy)]-diphthalonitrile **3** (0.5 g, 1.27 mmol), anhydrous $Zn(CH_3COO)_2$ (115.5 mg, 0.63 mmol) and 2-(dimethylamino)ethanol (DMAE) (3 ml) was irradiated in a microwave oven at 175 °C, 350 W for 12 min. After cooling to room temperature the reaction mixture was refluxed with ethanol to precipitate the product which was filtered off. The green solid product was washed with hot ethanol (40 ml) and dried in vacuo. This product is soluble in H_2SO_4 and hot pyridine. Yield: 747 mg (69%), mp > 300 °C. Elemental analyses (for imide end groups) $(C_{92}H_{56}N_{16}O_{16}Zn)n$ $(1706.93)_n$ calcd: C, 64.73; H, 3.30; N, 13.12; Zn, 3.83%; found: C, 64.98; H, 3.61; N, 12.81; Zn, 4.04. IR (KBr tablet) ν_{max}/cm^{-1}: 3411 (imide N–H), 3098 (Ar-H), 2928–2851 (Aliph. C–H), 1778 (sym. C=O), 1727 (asym. C=O), 1598 (C=N), 1528, 1345, 1288 (Ar-O–C), 1185, 1130. UV–vis (pyridine): λ_{max}/nm: $[(10^{-5}\varepsilon\ dm^3\ mol^{-1}\ cm^{-1})]$: 313 (4.98), 368 (4.92), 632 (4.52), 692 (5.13).

*3.2.5. Co(II)-containing polymer (**7**)*

A mixture of 4,4′-[pyridine-2,6-diylbis(methyleneoxy)]-diphthalonitrile **3** (0.5 g, 1.27 mmol), anhydrous $CoCl_2$ (81.7 mg, 0.63 mmol) and 2-(dimethylamino)ethanol (DMAE) (3 ml) was irradiated in a microwave oven at 175 °C, 350 W for 12 min. After cooling to room temperature the reaction mixture was refluxed with ethanol to precipitate the product which was filtered off. The green solid product was washed with hot ethanol (45 ml) and dried in vacuo. This product is soluble in H_2SO_4 and hot pyridine. Yield: 647 mg (60%), mp > 300 °C. Elemental analyses (for imide end groups) $(C_{92}H_{56}N_{16}O_{16}Co)n$ $(1700.49)_n$ calcd: C, 64.98; H, 3.31; N, 13.17; Co, 3.46%; found: C, 64.66; H, 3.67; N, 13.49; Co, 3.80. IR (KBr tablet) ν_{max}/cm^{-1}: 3417 (imide N–H), 3087 (Ar-H), 2912–2846 (Aliph. C–H), 1779 (sym. C=O), 1726 (asym. C=O), 1596 (C=N), 1522, 1456, 1336, 1256 (Ar-O–C), 1086. UV–vis (pyridine): λ_{max}/nm: $[(10^{-5}\varepsilon\ dm^3\ mol^{-1}\ cm^{-1})]$: 252 (5.15), 365 (5.13), 623 (4.50), 689 (5.19).

*3.2.6. Cu(I)-containing polymer (**8**)*

A mixture of 4,4′-[pyridine-2,6-diylbis(methyleneoxy)]-diphthalonitrile **3** (0.5 g, 1.27 mmol), anhydrous CuCl (62.3 mg, 0.63 mmol) and 2-(dimethylamino)ethanol (DMAE) (3 ml) was irradiated in a microwave oven at 175 °C, 350 W for 12 min. After cooling to room temperature the reaction mixture was refluxed with ethanol to precipitate the product which was filtered off. The green solid product was washed with hot ethanol (40 ml) and dried in vacuo. This product is soluble in H_2SO_4 and hot pyridine. Yield: 768 mg (71%), mp > 300 °C. Elemental analyses (for imide end groups) $(C_{92}H_{56}N_{16}O_{16}Cu)n$ $(1705.10)_n$ calcd: C, 64.80; H, 3.31; N, 13.14; Cu, 3.72%; found: C, 65.22; H, 3.66; N, 13.47; Cu, 3.38. IR (KBr tablet) ν_{max}/cm^{-1}: 3415 (imide N–H), 3082 (Ar-H), 2923–2846 (Aliph. C–H), 1770 (sym. C=O), 1703 (asym. C=O), 1596 (C=N), 1530, 1456, 1339, 1275 (Ar-O–C), 1185, 1141, 1072. UV–vis (pyridine): λ_{max}/nm: $[(10^{-5}\varepsilon\ dm^3\ mol^{-1}\ cm^{-1})]$: 275 (5.06), 348 (5.02), 618 (4.48), 688 (5.16).

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