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Synthesis, characterization and thermal behaviour of novel phthalocyanines bearing chalcone groups on peripheral positions

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A new nickel, zinc, cobalt, copper and metal-free phthalocyanine was synthesized by nucleophilic aromatic substitution reaction of (E)-3-(3-hydroxyphenyl)-1-phenylprop-2-en-1-one with 4-nitrophthalonitrile and cyclotetramerisation of (E)-4-(3-(3-oxo-3-phenylprop-1-enyl)phenoxy) phthalonitrile. The new compounds were characterized by a combination of IR, ^{1}H NMR, ^{13}C NMR, UV-Vis, elemental analysis and MS spectral data. The thermal stabilities of the phthalocyanine compounds were determined by thermogravimetric analysis.

Keywords: Phthalocyanine; Macrocyclic; Phthalonitrile; Chalcone; Thermogravimetric Analysis.

INTRODUCTİON

Phthalocyanines (Pcs) form nowadays an important group of organic compounds that belong to the most studied subjects of organic functional materials [1]. However, although no phthalocyanines have been identified in the nature yet, they were some of the first macrocycles that were synthesized and used as model compounds to mimic the biologically important porphyrines [2, 3]. Microwave irradiation is an important improvement in the synthesis of metallophthalocyanines. The most important industrial application of phthalocyanines is the formation of colored complexes with metal cations that are used as highly stable pigments and dyes [4]. In recent years, the applications of metallophthalocyanine (MPc) complexes have expanded to areas such as photosensitizers in photodynamic therapy, photoconducting agents in photocopying machines and electrocatalysts. In addition, they can find commercial applications as: photovoltaic materials in solar cells [5-7], systems for fabrication of light emitting diodes (LED) [8, 9], liquid crystalline [10] and non-linear optical materials [11, 12], sensitizers for photodynamic (PDT) cancer therapy [13, 14], photoconductors in xerography [15], dyes at recording layers for CD-R and DVD-R optical storage discs [16], as well as diverse catalytic systems [17, 18].

In medicine, phthalocyanines have been found to have applications as phototoxic drugs for photodynamic therapy [19-22]. Chalcones belong to the largest class of plant secondary metabolites, which, in many cases, serve in plant defense mechanisms to counteract reactive oxygen species (ROS) in order to survive and prevent molecular damage and damage by microorganisms, insects, and herbivores. They are known to possess antioxidant character of different extents [23].

Closed shell diamagnetic ions such as Zn^{2+}, Al^{3+} and Ga^{3+} give phthalocyanine complexes with both high triplet yields and long lifetimes of the excited triplet state. Thus, these complexes are expected to exhibit strong photochemical and photodynamic activities due to a higher efficiency in generating reactive oxygen species than porphyrins [19].

Some studies showed that a convenient route for the synthesis of metal-free phthalocyanine (H_2Pc) from phthalonitrile and metallophthalocyanines from phthalonitrile, phthalimide and phthalic anhydride using hexamethyldisilazane (HMDS). In spite of the potential utility of this approach, reaction times necessary for these reactions are very long at relatively high temperature. For example, the reaction times are 10-24 h at 100-150 °C when metal-free phthalocyanine is synthesized from phthalonitrile, 10-48 h at 150 °C when metallophthalocyanines are synthesized from phthalimide or phthalic anhydride and 10-12 h at 100-125 °C in the case of phthalonitrile [24, 25].

Microwave-promoted organic reactions are well known as environmentally benign methods that can accelerate a great number of chemical processes. In particular, the reaction time and energy input are supposed to be mostly reduced in the reactions that are run for a long time at high temperatures under conventional conditions [26].

In this article, we describe the synthesis and characterization of metal-free phthalocyanine **4** in 1.8-diazabicyclo[5.4.0]undec-7-ene (DBU) and *n*-

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pentanol in a Schlenk tube under N_2 and metallophthalocyanines **5-8** by microwave irradiation.

EXPERİMENTAL

All reactions were carried out under dry nitrogen atmosphere using standard Schlenk techniques. The IR spectra were recorded on a Perkin Elmer 1600 FTIR spectrophotometer, using potassium bromide pellets. ^1H and ^{13}C NMR spectra were recorded on a Varian Mercury 200 MHz spectrometer in $CDCl_3$, and chemical shifts are reported (δ) relative to Me_4Si as internal standard. Mass spectra were measured on a Varian 711 and VG Zapspec spectrometer. UV-visible absorpion spectra were measured by a Unicam UV-visible spectrometer. Melting points were measured on an Electrothermal apparatus. Domestic oven (Arçelik MD 823, 350 W) was used for the synthesis of metallophthalocyanines. A Seiko II Exstar 6000 thermal analyzer was used to record the DTA curves under nitrogen atmosphere with a heating rate of 20 ºC min^{-1} in the temperature range of 30 to 900 ºC using platinum crucibles. Domestic oven was used for all syntheses of phthalocyanines.

Synthesis of (E)-4-(3-(3-oxo-3-phenylprop-1-enyl)phenoxy)phthalonitrile (3)

(E)-3-(3-hydroxyphenyl)-1-phenylprop-2-en-1-one (**1**) **[**31]. (1 g, 4.46 mmol) was dissolved in dry DMF (15 ml) under N_2 atmosphere and 4-nitrophthalonitrile **(2)** (0.773 g, 4.46 mmol) was added to the solution. After stirring for 10 min finely ground anhydrous K_2CO_3 (3.07 g, 22.3 mmol) was added portionwise within 2 h under efficient stirring. The reaction mixture was then stirred under N_2 at 50 °C for 72 h. Then the solution was poured into ice-water (100 ml) and was stirred in the course of 1 day. The solid product was filtered, washed with water and dried *in vacuo* over P_2O_5. The product was recrystallized from ethanol. Yield: 1.19 g (76 %), mp: 132 °C; ^1H NMR ($CDCI_3$, 200 MHz) δ: 8.02-7.10 (m, Ar-H, olefinic C-H) ; ^{13}C NMR ($CDCl_3$, 100 MHz) δ: 190.29, 161.66, 154.34, 143.05, 138.07, 137.97, 135.80, 133.43, 131.53, 129.00, 128.79, 126.65, 123.84, 122.61, 121.89, 121.76, 120.25, 117.98, 109.43, 115.58, 115.14; IR (KBr) υ: 3076, 2926-2872, 2227, 1668, 1596, 1490, 1314, 1283, 1250, 1229, 1145, 1019, 844, 781, 688; MS (ES$^+$) *m/z*: 350 $[M]^+$. Anal. calcd for $C_{23}H_{14}N_2O_2$: C 78.84, H 4.03, N 8.00; found C 78.54, H 4.06, N 8.02.

Preparation of metal-free phthalocyanine (4)

(E)-4-(3-(3-oxo-3-phenylprop-1-enyl)phenoxy) phthalonitrile **(3)** (300 mg, 0,86 mmol), DBU (five drops) and dry *n*-pentanol (4 ml) were added in a Schlenk tube. The mixture was heated and stirred at 160 °C for 24 h under N_2. Thereafter the reaction mixture was cooled to 30 °C and precipitated by adding ethanol. The solid product was filtered and washed with ethanol. The green solid product was chromatographed on silica gel with chloroform/methanol (9:1) as an eluent. Yield: 48 mg (16 %), UV-vis (chloroform) λ_{max}: 705(2.85), 669(2.79), 642(2.45), 605(2.25), 396(2.35), 341(2.68) nm; ^1H NMR ($CDCI_3$, 200 MHz) δ: 7.73-6.72(m, Ar-H, olefinic C-H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ: 176.12, 172.22, 172.74, 163.30, 162.42, 157.28, 154.46, 146.21, 134.14, 131.12, 130.86, 127.94, 126.42, 124.31, 123.64, 122.89, 118.26, 116.42, 114.18; IR (KBr) υ: 3426, 3060, 2923-2853, 1662, 1571, 1464, 1376, 1269, 1119, 852, 750, 692; MS (ES$^+$) *m/z*: 1404 $[M+1]^+$. Anal. calcd for $C_{92}H_{58}N_8O_8$ C 78.66, H 4.13, N 7.98, found C 78.62, H 4.11, N 7.96.

Nickel (II) phthalocyanine (5)

A mixture of (E)-4-(3-(3-oxo-3-phenylprop-1-enyl)phenoxy) phthalonitrile **(3)** (200 mg, 0,57 mmol), anhydrous metal salt $NiCl_2$ (18.5 mg, 0.14 mmol), and 2-(dimethylamino)ethanol (3 ml) was irradiated in a microwave oven at 175 °C, 350 W for 8 min. After cooling to room temperature the reaction mixture was refluxed with ethanol to precipitate the product which was filtered off and dried *in vacuo* over P_2O_5. The obtained green solid product was purified by column chromatography on silica gel with chloroform-methanol (5:1) as an eluent. Yield: 69 mg (33 %), UV-vis (chloroform) λ_{max}: 678(2.88), 653(2.51), 436(2.57), 353(3.21) nm; ^1H NMR ($CDCI_3$, 200 MHz) δ: 7.90-6.32 (m, Ar-H, olefinic C-H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ: 199.44, 173.32, 168.71, 162.03, 156.41, 143.80, 142.99, 139.73, 138.06, 133.21, 130.85, 128.79, 125.60, 124.29, 122.86, 121.81, 119.99, 113.81, 113.08, 112.27, 111.86, 110.24,107.59; IR (KBr) υ: 3060, 2927-2846, 1665, 1577, 1473, 1446, 1330, 1236, 1147, 1093, 1017, 977, 772, 689; MS (ES$^+$) *m/z*: 1460 $[M]^+$; Anal. calcd for $C_{92}H_{56}N_8O_8Ni$: C 75.60, H 3.83, N 7.67; found C 75.63, H 3.81, N 7.69.

Zinc (II) phthalocyanine (6)

A mixture of (E)-4-(3-(3-oxo-3-phenylprop-1-enyl)phenoxy) phthalonitrile **(3)** (200 mg, 0,57 mmol), anhydrous metal salt $Zn(CH_3COO)_2$ (29 mg, 0.14 mmol), and 2-(dimethylamino)ethanol (3 ml) was irradiated in a microwave oven at 175 °C, 350 W for 6 min. After cooling to room temperature the reaction mixture was refluxed with ethanol to precipitate the product which was filtered off and dried *in vacuo* over P_2O_5. The obtained green solid product was purified from the column chromatography on silica gel with chloroform-methanol (6:1) as an eluent. Yield: 107 mg (51 %), UV-vis (chloroform) λ_{max}: 682(3.12), 651(2.45), 400(2.99), 353(3.16) nm; ^{1}H NMR ($CDCI_3$, 200 MHz) δ: 7.98-6.87 (m, Ar-H, olefinic C-H) ; ^{13}C NMR ($CDCl_3$, 100 MHz) δ: 186.44, 174.23, 171.02, 166.88, 161.54, 157.44, 151.13, 146.97, 141.85, 132.72, 130.16, 129.58, 128.78, 124.88, 121.42, 120.13, 119.17, 118.42, 110.02, 105.00; IR (KBr) υ: 3061, 2927-2851, 1664, 1578, 1484, 1393, 1314, 1236, 1179, 1089, 1045, 977, 880, 773; MS (ES$^+$) *m/z*: 1467 [M+1]$^+$; Anal. calcd for $C_{92}H_{56}N_8O_8Zn$: C 75.26, H 3.82, N 7.63; found C 75.24, H 3.86, N 7.62.

Cobalt (II) phthalocyanine (7)

A mixture of (E)-4-(3-(3-oxo-3-phenylprop-1-enyl)phenoxy) phthalonitrile **(3)** (200 mg, 0,57 mmol), anhydrous metal salt $CoCl_2$ (18.5 mg, 0.14 mmol) and 2-(dimethylamino)ethanol (3 ml) was irradiated in a microwave oven at 175 °C, 350 W for 8 min. After cooling to room temperature the reaction mixture was refluxed with ethanol to precipitate the product which was filtered off and dried *in vacuo* over P_2O_5. The obtained green solid product was purified by column chromatography on silica gel with chloroform-methanol (5:1) as an eluent. Yield: 95 mg (46 %), UV-vis (chloroform) λ_{max}: 672(2.75), 618(2.32), 302(3.16) nm; IR (KBr) υ: 3061, 2927-2846, 1665, 1577, 1473, 1329, 1237, 1179, 1095, 1017, 977, 881, 773, 689; MS (ES$^+$) *m/z*: 1460 [M]$^+$; Anal. calcd for $C_{92}H_{56}N_8O_8Co$: C 75.59, H 3.83, N 7.67; found: C 75.94, H 3.60, N 7.31.

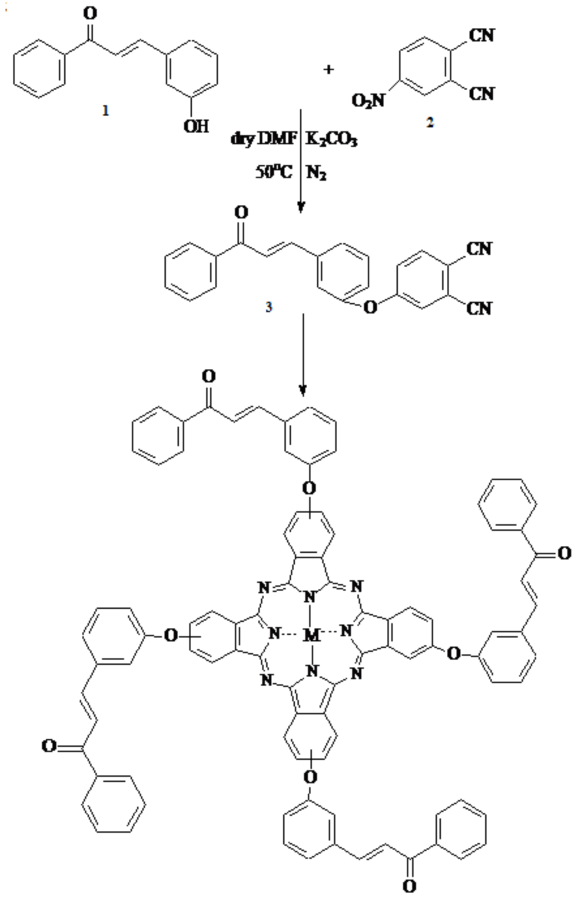
Copper (II) phthalocyanine (8)

A mixture of (E)-4-(3-(3-oxo-3-phenylprop-1-enyl)phenoxy) phthalonitrile **(3)** (200 mg, 0,57 mmol), anhydrous metal salt $CuCl_2$ (19.1 mg, 0.14 mmol) and 2-(dimethylamino)ethanol (3 ml) was irradiated in a microwave oven at 175 °C, 350 W for 5 min. After cooling to room temperature the reaction mixture was refluxed with ethanol to precipitate the product which was filtered off and dried *in vacuo* over P_2O_5. The obtained green solid product was purified by column chromatography on silica gel with chloroform-methanol (6:1) as an eluent. Yield: 73 mg (35 %), UV-vis (chloroform) λ_{max}: 684(2.83), 618(2.42), 298(3.17) nm; IR (KBr) υ: 3061, 2928-2846, 1664, 1577, 1479, 1446, 1314, 1237, 1146, 1092, 1017, 977, 773, 690; MS (ES$^+$) *m/z*: 1466 [M+1]$^+$; Anal. calcd for $C_{92}H_{56}N_8O_8Cu$: C 75.35, H 3.82, N 7.64; found C 75.33, H 3.85, N 7.62.

RESULTS AND DİSCUSSİON

Synthesis of (E)-4-(3-(3-oxo-3-phenylprop-1-enyl)phenoxy) phthalonitrile **3** was carried out by the reaction of 4-nitrophthalonitrile **(2)** and (E)-3-(3-hydroxyphenyl)-1-phenylprop-2-en-1-one **(1)** in the presence of K_2CO_3 in dry DMF. Final purification of the phthalonitrile derivative **3** by recrystallization afforded **3** with satisfactory elemental analysis and mass spectral data. In the ^{1}H-NMR spectrum of the phthalonitrile derivative **3**, the OH group of compound **1** disappeared as expected. The disappearance of OH and the presence of C≡N functional group at 2227 cm^{-1} in the IR spectrum of compound **3** confirm the formation of the desired compound **3**. The ^{13}C-NMR spectrum of the phthalonitrile derivative **3** indicated the presence of nitrile carbon atoms at 115.58, 115.14 (C≡N) ppm.

The metal-free phthalocyanine **4** was synthesized by heating a mixture of phthalonitrile derivative **3**, DBU and dry n-pentanol in a Schlenk tube. The IR spectrum of the metal-free phthalocyanine **4** shows 3426 cm^{-1} (N-H) vibrations. The disappearance of the C≡N stretching vibration in the IR spectrum of phthalonitrile derivative **3** suggested the formation of compound **4**. In the ^{1}H-NMR spectrum of this compound **4** the inner core protons of Pc-2H could not be observed due to strong aggregation of molecules [27]. The mass spectrum of this compound at (m/z): 1404 [M+1]$^+$ supports the proposed formula for this structure. The elemental analysis confirmed the structure of compound **4** (Fig. 1). The transition metal complexes were prepared from the phthalonitrile derivative **3** and the corresponding metal salt by microwave irradiation in 2-(dimethylamino) ethanol at 175°C, 350 W. The IR spectra of the metal-free compound **4** and the metallophthalocyanine compounds **5-8** are very similar. The significant difference is the presence of (N-H) vibrations of the inner phthalocyanine core which are assigned to a weak band at 3426 cm^{-1} in the metal-free molecule. These bands disappear in the spectra of the metallophthalocyanine complexes. The disappearance of the C≡N stretching vibration in the IR spectra of compound **3** suggested the formation of compounds **5-8**. The NMR characteristics of the nickel compound 5 and zinc the compound 6 were similar to those of the precursor dicyano compound **3** and the metal-free phthalocyanine compound **4**. In the mass spectrum of Ni, Zn, Co and Cu phthalocyanines, the presence of molecular ion peaks at (m/z): 1460 $[M]^+$, 1467 $[M+1]^+$, 1460 $[M+1]^+$, 1466 $[M+1]^+$ respectively, confirmed the proposed structures (Fig. 1). The elemental analyses of the metallophthalocyanines 5-8 were satisfactory.



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

Fig. 1. Synthesis of metal-free phthalocyanine and metallophthalocyanines

The UV-vis absorption spectra of compounds **4-8** in chloroform at room temperature are shown in Figs. 2 and 3. In general, phthalocyanines show typical electronic spectra with two strong absorption regions, one in the UV region at about 300-350 nm related to the B band and the other in the visible region at 600-700 nm related to the Q band. The split Q bands in compound **4**, which are characteristic for metal-free phthalocyanines, were observed at λ_{max}= 705, 669 nm, respectively. These intense Q bands indicate monomeric species, as species with D_{2h} symmetry show two intense absorption bands around 700 nm [28]. Such split Q band absorptions are due to the π→π* transition from the HOMO to the LUMO of the phthalocyanine ring related to the fully conjugated 18π electron system. The presence of strong absorption bands in compound **4** in the near UV region at λ_{max}= 341 nm also shows Soret region B bands which have been ascribed to the deeper π→π* levels of LUMO transations [29].

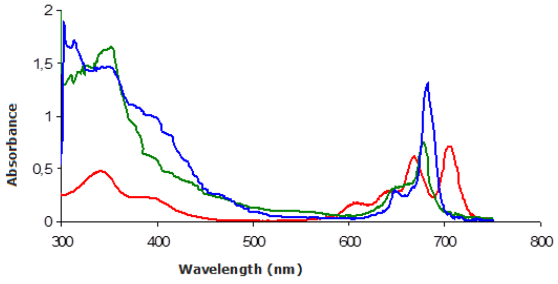


Fig. 2. UV-vis spectra of H_2Pc(—),NiPc(—) and ZnPc(—) complexes

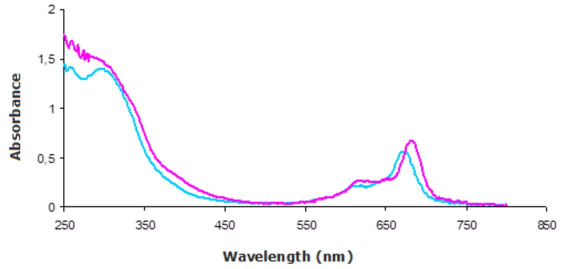


Fig. 3. UV-vis spectra of CoPc(—) and, CuPc(—) complexes

The UV-vis absorption spectra of the metallophthalocyanines **5-8** show intense Q band absorptions at λmax = 678, 682, 672, 684 nm, respectively. The single Q bands in the metalloderivatives and the split form in their metal-free derivatives are characteristic [30].

The thermal behaviour of the metallophthalocyanines were investigated by TG/DTA. Although the thermal stabilities of phthalocyanines are well known, the phthalocyanine compounds are not stable above 292 °C. The initial and main decomposition temperatures are given in Table 1. The initial decomposition temperatures decreased in the order: 8>6 >7 >5.

Table 1. Thermal properties of the metallophthalocyanines

Compound	M	Initial decomposition temperature in °C	Main decomposition temperature in °C
5	Ni	292	336
6	Zn	308	391
7	Co	300	399
8	Cu	357	393

CONCLUSİON

In this work, we describe the synthetic procedure and characterization of new metal-free and metallophthalocyanines. A dinitrile monomer was synthesized by nucleophilic aromatic substitution of (E)-3-(3-hydroxyphenyl)-1-phenylprop-2-en-1-one onto 4-nitrophthalonitrile.

The newly developed methods for the synthesis of phthalocyanine are easier, more economical, and less time-consuming. The microwave energy facilitates the synthesis. In addition, the thermal properties of the new phthalocyanines were examined by thermogravimetric analysis. Phthalocyanines are known as compounds of high thermal stability. So this property enables the use of phthalocyanines as technological materials.

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СИНТЕЗА, ХАРАКТЕРИЗИРАНЕ И ТЕРМИЧНО ПОВЕДЕНИЕ НА НОВИ ФТАЛОЦИАНИНИ, НОСЕЩИ ХАЛКОНОВАТА ГРУПИ НА ПЕРИФЕРНИ ПОЗИЦИИ

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(Резюме)

Нови фталоцианини на никел, цинк, кобалт, мед и некомплексен фталоцианин бяха синтезирани чрез реакция на нуклеофилно ароматно заместване на (Е) -3 - (3-хидроксифенил) -1-фенилпроп-2-ен-1-он с 4-нитрофталонитрил и циклотетрамеризация на (Е) -4- (3- (3-оксо-3-фенилпроп-1-енил) фенокси) фталонитрил. Новите съединения са охарактеризирани чрез комбинация от ИЧ, 1Н ЯМР, 13С ЯМР, УВ спектроментрия, елементен анализ и масспектрални данни. Термичните стабилности на фталоцианинови съединения бяха определени чрез термогравиметричен анализ.