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The syntheses, characterization and fluorescence spectra of novel, octakis(alkylthiophthalocyanato) nickel(II) and palladium(II) complexes

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The syntheses, characterization and fluorescence studies of several {octakis(alkylthio)phthalocyanato} palladium(II) and nickel(II) complexes are presented. The absorption spectra of some of the complexes showed extra peaks which are attributable to non-planar distortion, the extent of which, was found to dependent on alkyl chain length. The fluorescence excitation spectra of the nickel(II) derivatives were not in agreement with their absorption spectra, owing to structural changes upon excitation. Fluorescence quantum yields were very low (<1%) for all complexes as a consequence of the open-shell electronic structures of nickel(II) and palladium(II).

1. Introduction

Phthalocyanines (Pcs) and their metal complexes (MPcs) are among the most studied classes of compounds because of their various applications and their chemical and thermal stability. The optical and electrochemical activities which are characteristic of the array of Pc derivatives also hold great promise for a wide range of commercial applications. Chemical sensor development [1,2], fibrous assemblies [3–6], nanostructured material preparation and artificial light antenna for solar energy conversion [7–11], are among many areas of research on these versatile dyes.

The application capabilities of these compounds are dependent on their molecular composition. Factors such as the nature of substituents and type of metal in the phthalocyanine cavity play a role in this regard. The molecular architecture such as the position of the substituents is also important in controlling some of the Pc properties [12–18].

Introduction of appropriate functional groups on the macrocycle enhances the solubility by causing substantial disruption of the interactions between the phthalocyanine rings [19,20]. Phthalocyanines with either alkyl or alkoxy groups or polyoxyethylene [21,22] substituent have shown improved solubility.

The photophysical properties of various metal Pcs have been extensively studied however reports concerning alkylthio substituted Pd(II) and Ni(II) Pcs are scarce. Even though there is a growing interest in open-shell metal Pcs for application in the areas of catalysis [23,24] and electrochemistry [25], the study of their physicochemical properties is of importance. Octasubstitution of PdPc with butoxyl groups at the α-position resulted in a macrocycle with a high triplet quantum yield [26], while tetrasubstitution at the β-position with *t*-butyl groups gave a typical quantum yield [27]. Introduction of alkylthio substituents of different chain lengths into the MPc ring might affect spectroscopic properties, fluorescence quantum yields and lifetimes; hence it is desirable to carry out studies on the effects of substituents on these parameters. This work reports on the synthesis and investigation of fluorescence properties of nickel(II) and palladium(II) octakis(alkylthio) phthalocyanines. The synthesis of Ni octakis (dodecylthio) phthalocyanine (**6c**) has been reported [28], but no work has been reported on its fluorescence behaviour. Octylthio and dodecylthio phthalocyanine complexes of Cu have been reported [29] as well as non-peripherally substituted octylthio ZnPc derivatives [30]. There have been no reports of the Pd alkyl derivatives of the kind reported here and for NiPc derivatives the reports are limited. The fluorescence behaviour of the NiPc and PdPc derivatives are also scarce.

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2. Experimental

2.1. Materials

1-Pentanol, 1-pentanethiol, dimethylsulphoxide (DMSO), 1.8-diazabicyclo[5, 4, 0] undec-7-ene (DBU), cerium chloride, dichloromethane (DCM) and palladium(II) chloride were purchased from Sigma–Aldrich. Chloroform and 1-chloronaphthalene were purchased from Merck. Chromatography was performed on silica gel 60 (0.04–0.063 mm). 1,2-Dichloro-4,5-dicyanobenzene (**3**) [31,32], 1,2-bis(octylthio)-4,5-dicyanobenzene (**4b**) and 1,2-bis(dodecylalthio)-4,5-dicyanobenzene (**4c**) were synthesized as in literature [28,33]. The synthesis of **6c** has been reported before [28].

2.2. Equipment

UV/Vis and fluorescence spectra were recorded on a Varian Cary 500 UV/Vis/NIR and Varian Eclipse spectrophotometer respectively, ^{1}H NMR spectra were recorded using a Bruker EMX 400 MHz NMR spectrometer. IR spectra (KBr pellets) were recorded on a Perkin–Elmer spectrum 2000 FTIR Spectrometer.

2.3. Synthesis

2.3.1. 1,2-Bis(pentylthio)-4,5-dicyanobenzene (*4a*)

1-Pentanethiol (2.00 g, 20 mmol)] was dissolved in anhydrous dimethylsulfoxide (DMSO) (15 ml) under nitrogen and 1,2-dichloro-4,5-dicyanobenzene (**3**) (2 g, 7.6 mmol) was added. After stirring for 15 min, finely ground anhydrous potassium carbonate (6 g, 43.4 mmol) was added portionwise within 2 h with vigorous stirring. The reaction mixture was stirred under nitrogen at room temperature for 12 h. Then water (30 ml) was added and the aqueous phase extracted with chloroform (3 × 20 ml). The combined extracts were treated first with sodium carbonate solution (5%), then with water and the solvent was evaporated and the product was crystallized from EtOH. Yield: 1.9g(79%); ^{1}H NMR (400 MHz, $CDCl_3$) $\boldsymbol{\delta_H}$/ppm: 7.72 (2H, s, Ar–H), 3.05–3.01 (4H, t, S–CH_2), 1.84–1.75 (4H, m, –CH_2), 1.59–1.48 (4H, t, –CH_2), 1.42–1.38 (4H, m, –CH_2), 0.97–0.94 (6H, t, –CH_3); [IR (KBr pellets) ν_{max}/cm^{-1}]: 3467,3094, 3022, 2933, 2871, 2574, 2236(C≡N), 1825, 1573, 1469, 1352, 1226, 1127, 916, 738(C–S), 687, 531.

2.3.2. 2,3,9,10,16,17,23,24-Octakis(pentylthiophthalocyaninato) palladium(II) (*5a*)

A mixture of compound **4a** (0.361 g, 1 mmol) and $PdCl_2$ (0.08 g, 0.45 mmol) was heated under reflux in pentanol (5 ml) in the presence of DBU (1.66 ml, 1 mmol) with stirring for 1 h in a nitrogen atmosphere. After cooling, the dark green product was precipitated with methanol, filtered and then purified using silica gel packed column chromatography with DCM as eluent. Yield: 0.19 g (50%), UV/Vis [(1-chloronaphthalene/λ_{max}/nm, (log ε))] 744 (4.13), 698 (5.14), 683 (4.6),425 (4.55). $C_{72}H_{96}N_8PdS_8$:Cald.: C, 60.20; H, 6.74; N,7.80%; Found, C, 59.42; H, 7.11; N,7.01%; ^{1}H NMR (400 MHz, $CDCl_3$) $\boldsymbol{\delta_H}$/ppm: 8.19 (8H, s, Ar–H), 3.40 (16H, m, S–CH_2), 1.14–1.09 (48H, m, –$(CH_2)_3$), 0.91–0.88 (24H, m, –CH_3); [IR (KBr pellets) ν_{max}/cm^{-1}]: 2595, 2928, 2858, 1737, 1603, 1510, 1465, 1413, 1379, 1265, 1142, 1075, 968, 747(C–S).

2.3.3. 2,3,9,10,16,17,23,24-Octakis(octylthiophthalocyaninato) palladium(II) (*5b*)

The same method used for **5a** was employed for the synthesis of **5b**, except **4b** was employed instead of **4a**. The amounts of reagents were: **4b** (0.42 g, 1 mmol) and $PdCl_2$ (0.08 g, 0.45 mmol). Yield: 0.26 g (60%). UV/Vis [(1-chloronaphthalene/λ_{max}/nm, (log ε)] 698 (4.88), 673 (4.25), 625 (4.31), 412 (4.50) $C_{96}H_{144}N_8PdS_8$:Cald.: C, 59.34; H, 7.40; N,5.70%; Found, C, 58.02; H, 8.06; N,4.02%. ^{1}H NMR (400 MHz, $CDCl_3$) $\boldsymbol{\delta_H}$/ppm: 8.21 (8H, s, Ar–H), 3.41 (16H, m, S–CH_2), 1.60–1.32 (120H, m, –$(CH_2)_6$–CH_3)); [IR (KBr pellets) ν_{max}/cm^{-1}]: 2962, 2930, 2858, 1466, 1414, 1381, 1264, 1143, 1078, 969, 874, 747(C–S).

2.3.4. 2,3,9,10,16,17,23,24-Octakis(dodecylthiophthalocyaninato) palladium(II) (*5c*)

The same method used for **5a** was employed for the synthesis of **5c**, except **4c** was employed instead of **4a**. The amounts of reagents were: **4c** (0.53 g, 1 mmol) and $PdCl_2$ (0.08 g, 0.45 mmol). Yield: 0.30 g (55%). UV/Vis [(1-chloronaphthalene/λ_{max}/nm, (log ε)] 698 (4.92), 673 (4.25), 625 (4.29), 412 (4.50). $C_{130}H_{212}N_8PdS_8$:Cald.: C, 64.43; H,8.70; N,4.60%. Found, C, 62.97; H,9.44; N,3.13%; ^{1}H NMR (400 MHz, $CDCl_3$) $\boldsymbol{\delta_H}$/ppm:8.35 (8H, s, Ar–H), 3.40 (16H, m, S–CH_2), 1.25–0.75 (184H, m, –$(CH_2)_{10}$–CH_3)); [IR (KBr pellets) ν_{max}/cm^{-1}]: 2926, 2856, 1731, 1468, 1379, 1266, 1108, 998, 818, 749(C–S), 649.

2.3.5. 2,3,9,10,16,17,23,24-Octakis(pentylthiophthalocyaninato) nickel(II) (*6a*)

The same method used for **5a** was employed for the synthesis of **6a**, except $NiCl_2$ was employed instead of $PdCl_2$. The amounts of reagents were: used with **4a** (0.361 g, 1 mmol) and $PdCl_2$ was replaced with $NiCl_2$. $6H_20$ (0.12 g, 0.5 mmol) that was preheated in the oven. Yield: 0.27 g (80%). UV/Vis [(1-chloronaphthalene/λ_{max}/nm, (log ε))] 725 (4.81), 697 (5.14), 665 (4.75), 628 (4.63, 424 (4.54) $C_{72}H_{96}N_8NiS_8$:Cald.: C,58.66; H,6.57; N,7.60%. Found, C, 57.81; H, 4.91; N, 9.96%. ^{1}H NMR (400 MHz, $CDCl_3$) $\boldsymbol{\delta_H}$/ppm: 8.23 (8H, s, Ar–H), 3.32 (16H, m, S–CH_2), 1.48–1.31 (72H, m, –$(CH_2)_3$–CH_3).; [IR (KBr pellets) ν_{max}/cm^{-1}]: 2930, 2860, 2235, 1604, 1465, 1421, 1411, 1350, 1200, 1077, 1023, 967, 956, 883, 788, 750(C–S), 658, 512.

2.3.6. 2,3,9,10,16,17,23,24-Octakis(octylthiophthalocyaninato) nickel(II) (*6b*)

The same method used for **5a** was employed for the synthesis of **6b**, except $NiCl_2$ was employed instead of $PdCl_2$ and **4b** used instead of **4a**, the amounts of reagents were: **4b** (0.42 g, 1 mmol) and $NiCl_2$. $6H_20$ (0.12 g, 0.5 mmol). Yield: 0.25 g (60%). UV/Vis [(1-chloronaphthalene/λ_{max}/nm, (log ε)] 707 (5.29), 679 (4.72), 634 (4.62), 424 (4.57). $C_{96}H_{144}N_8NiS_8$: Cald.: C, 66.83; H, 8.41; N,6.49% . Found, C, 66.67; H, 8.23; N,6.65%. ^{1}H NMR (400 MHz, $CDCl_3$) $\boldsymbol{\delta_H}$/ppm: 8.17 (8H, s, Ar–H), 3.36 (16H, m, S–CH_2), 1.62–1.39 (120H, m, –$(CH_2)_6$–CH_3)); [IR (KBr pellets) ν_{max}/cm^{-1}]: 2929, 2857, 1743, 1602, 1537, 1467, 1420, 1386, 1350, 1295, 1075, 970, 870, 787, 750(C–S), 691, 519.

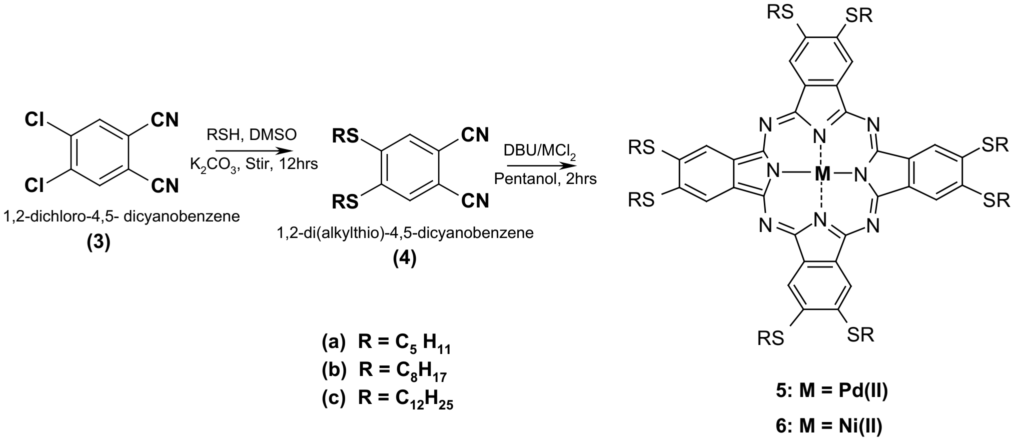
2.4. Photophysical properties

2.4.1. Fluorescence quantum yields

Fluorescence quantum yields (Φ_F) were determined by the comparative method, using Eq. (1) [34,35]:

$$\Phi_F = \Phi_F^{std} \frac{F \cdot A_{std} \cdot n^2}{F_{std} \cdot A \cdot n_{std}^2} \tag{1}$$

where F and F_{std} are the areas under the fluorescence curves of the MPc derivatives and the standard, respectively. A and A_{std} are the respective absorbances of the sample and standard at the excitation wavelengths (which was 0.05), and n and n_{std} are the refractive indices of the solvents used for sample and standard, respectively. ZnPc was employed as a standard in DMSO ($\Phi_F = 0.20$) [34].



Scheme 1. Synthesis of palladium and nickel octakis(pentylthio)phthalocyanines.

3. Results and discussion

3.1. Syntheses and spectral characterization

1,2-Dichlorophthalonitrile (**3**), was used to synthesize the three bis(alkythio)phthalonitriles (**4a**, **4b** and **4c**) through a base promoted nucleophilic aromatic displacement. The syntheses of **4b** and **4c** have been previously reported [28,33]. The reactions were carried out in DMSO at room temperature and gave yields of 79%, 76% and 71% for **4a**, **4b** and **4c** respectively. Octasubstituted phthalocyanines are prepared by cyclotetramerization of disubstituted phthalonitriles [32].

The syntheses of MPcs (**5a**, **5b**, **5c**, **6a**, **6b** and **6c**) were achieved by treating the corresponding phthalonitriles (**4a**, **4b** and **4c**) with palladium chloride in 1-pentanol for palladium phthalocyanine complexes (**5a**, **5b** and **5c**) and anhydrous nickel chloride in the same solvent for nickel phthalocyanine complexes (**6a**, **6b** and **6c**) (Scheme 1). The synthesis of **6c** has been previously reported [28].

Column chromatography with silica gel was employed to obtain the pure products. The MPc derivatives were characterized by UV/Vis, IR and NMR spectroscopies, and elemental analyses. The analyses were consistent with the predicted structures. It has been reported before that for large Pc molecules, elemental analyses often gives unsatisfactory results [36]. However, elemental analyses gave reasonable results confirming the identities of the complexes. The sharp peak for the C≡N vibrations in the IR spectra of phthalonitriles **4a**, **4b** and **4c** at 2235, 2233 and 2233 cm^{-1}, respectively, disappeared after conversion into phthalocyanines.

All six complexes showed vibrational peaks between 700 and 1000 cm^{-1} which are assigned to phthalocyanines skeletal vibrations [37]. The ^{1}H NMR spectra of MPc derivatives in CDCl$_3$ showed all the substituent and Pc ring protons observed in their respective regions. A singlet, assigned to Pc aromatic ring protons, appeared in the range 8.17–8.48 (integrating for 8 protons) in the ^{1}H NMR spectra **5** and **6**. A multiplet, assigned to the first methylene group belonging to the alkylthio side chains, appeared in the range 3.32–3.44 (integrating for 16 protons) in the ^{1}H NMR spectra **5** and **6**.

All the complexes were soluble in 1-chloronaphthalene, DCM, chloroform and toluene but they were highly aggregated in DCM, chloroform and toluene. Their insolubility in highly polar solvents like DMF and DMSO may be due to the presence of the highly non-polar alkyl chain.

3.2. Ground state electronic absorption

The ground state electron absorption spectra of **5a**–**c** and **6a**–**c** are shown in Figs. 1 and 2 respectively. For the PdPc derivatives (**5a**–**c**), the *Q*-band position is constant at 698 nm (Table 1), implying that the alkyl chain length does not significantly affect the *Q*-band position in these complexes. However for the NiPc derivatives, the *Q*-band positions for **6b** (707 nm) and **6c** (709 nm) are red-shifted relative to that of **6a** (696 nm). On the average, the *Q*-band positions for the NiPc complexes are red-shifted relative to those of the PdPc complexes. The spectra of the pentylthio-substituted derivatives (**5a** and **6a**) exhibit extra peaks at ~740 nm, in addition to the normal *Q*-maxima.

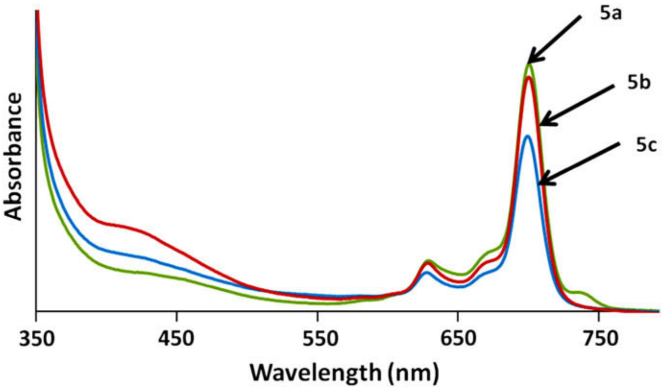


Fig. 1. Absorption spectra of complexes **5a**, **5b** and **5c** in 1-chloronaphthalene. Concentration ~8 × 10^{-6} mol dm^{-3}.

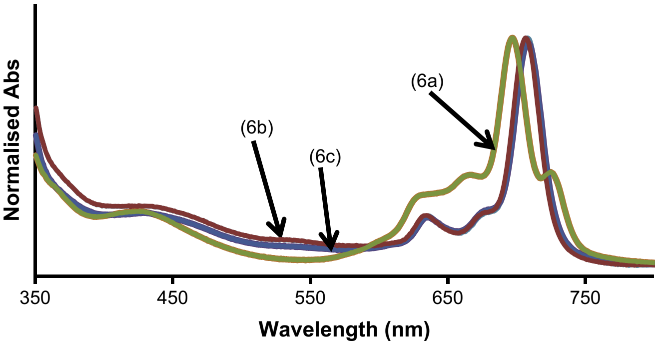


Fig. 2. Absorption spectra of complexes **6a**, **6b** and **6c** in 1-chloronaphthalene. Concentration ~8 × 10^{-6} mol dm^{-3}.

Table 1
UV/Vis spectral parameters of PdPc (**5a–c**) and NiPc (**6a–c**) derivatives in 1-chloronaphthalene.

MPc	Q_{abs}(nm)	Q_{ems}(nm)	Q_{exc}(nm)	Stokes' shift (nm)
5a	698	745	738	47
5b	698	707	701	9
5c	698	709	703	11
6a	696	730	695,725	34
6b	707	740	662,699	33
6c	709	730	727	21

The split in the *Q*-band for alkylthio derivatives of CuPc and NiPc has been observed and could not be explained in terms of aggregation or demetalation since an extra band was also observed for unmetallated derivatives [28]. The split in the *Q*-band was explained in terms of intermolecular interaction between the central metal ion of one molecule with the thio group of another [28]. The extent of the split depended on the solvent [28]. We propose the same type of interaction for the current complexes, with the extent of the interaction resulting in differences in the extent of splitting of the *Q*-bands of the complexes. Thus the splitting depends on the central metal and on the length of the chain. In the published work [28], the dodecylthio NiPc derivative showed the splitting, but in different solvents. For **6a**, there is split in the *Q*-band accompanied by a more blue shifted *Q*-band compared to **6b** and **6c**. The *Q*-band spectra of alkyl MPc complexes are not known to shift with change in the chain length [30,38,39]. The observed spectra may suggest some demetalation, but refluxing the solution of **6a** in chloronaphthalane in the presence of a nickel chloride did not change the spectra in Fig. 2. Also the spectra for **6a** in Fig. 2 do not match that of the unmetalated derivative.

As stated above, the complexes were highly aggregated in DCM, chloroform and toluene. It is also known that the degree of aggregation is increased by the presence of long alkylchains [40]. The complexes studied in this work are not aggregated in 1-chloronaphthalene at the concentrations used (hence this solvent was employed), as attested by their conformity to the Beer–Lambert law up to concentrations ~1.00 × 10^{-5} M (Fig. 3). Also, addition of surfactant, Triton X-100 brought about no observable spectral change. Again, the possibility of these complexes existing as mixtures of isomers is ruled out, as they all are octasubstituted. As a result, the observed band splitting will most likely have arisen from the intermolecular interactions discussed above. The scope of study on the complexes is limited by their aggregation in most solvents.

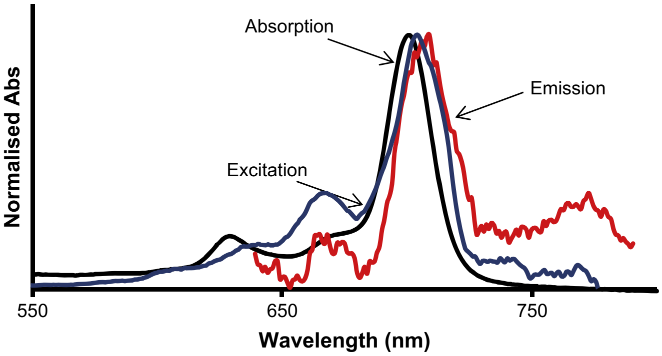


Fig. 4. Absorption and fluorescence (excitation and emission) spectra of **5c** in 1-chloronaphthalene. Excitation wavelength = 630 nm.

3.3. Fluorescence spectra and quantum yields

The absorption and fluorescence excitation spectra of the PdPc derivatives (**5b** and **5c**) are almost identical (Fig. 4), implying that the molecules absorbing species do not differ much from the fluorescing species; the fluorescence spectra are red-shifted with respect to the absorption spectra, (Fig. 4). The fluorescence excitation spectrum of the pentylthio-substituted PdPc derivative (**5a**) is different from the absorption spectrum, in a similar manner to NiPc derivatives, Fig. 5. For the NiPc derivatives, the fluorescence

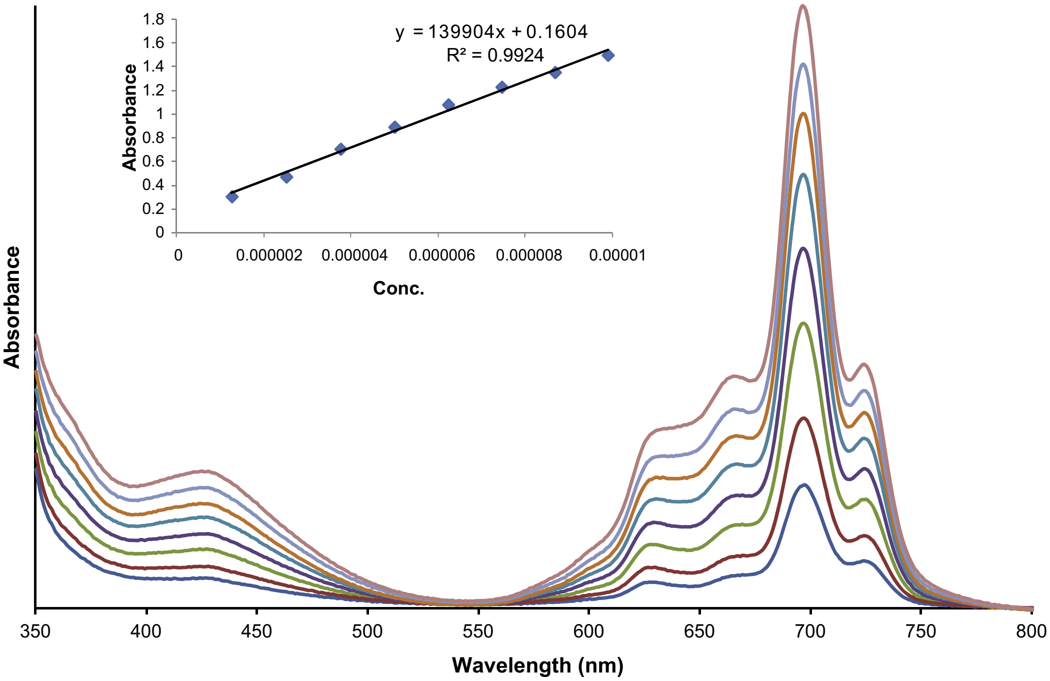


Fig. 3. Spectral changes accompanying dilution of **6a** in 1-chloronaphthalene. Inset: Beer–Lambert's plot for **6a** in 1-chloronaphthalene.

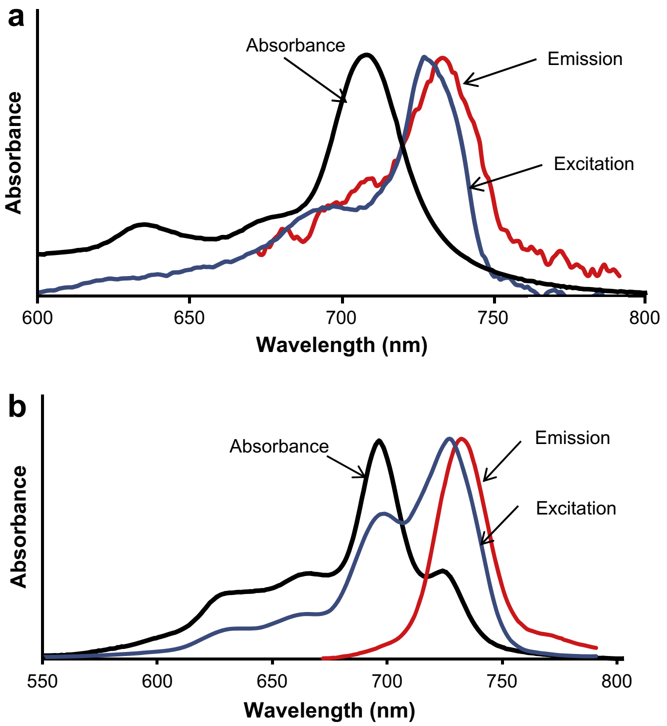


Fig. 5. Absorption and fluorescence (excitation and emission) spectra of (a) **6c** and (b) **6a** in 1-chloronaphthalene. Excitation wavelength = 630 nm.

excitation spectrum of **6c** (Fig. 5a) is red-shifted by about 20 nm relative to that of the absorption spectrum, suggesting a change in geometry upon excitation. In the case of **5a**, **6a** and **6b** (Fig. 5b), the absorption and fluorescence excitation spectra are not similar; although there is almost a peak-to-peak matching, the intensities are reversed (Fig. 5b), which could be due to excitonic intensity borrowing and exchange between the two major transitions.

The fluorescence quantum yield (Φ_F) values of **5a**–**c** and **6a**–**c** in 1-chloronaphthalene are predictably low (<0.01). These low values are attributed to the enhancement of intersystem crossing (spin–orbit coupling) due to the heavy atom effect of the chlorine in the solvent molecule. It has also been reported that distortion in MPc complexes leads to low Φ_F values [41]. Again low Φ_F values are expected for NiPc and PdPc derivatives because of the open-shell nature of the complexes. There is also potential for considerable d–π interactions to occur, which would quench the excited singlet state, resulting in low fluorescence quantum yield. The weak fluorescence emitted by these complexes could also have arisen from their non-planarity. It is known that non-planar porphyrins have significantly lower Φ_F values than their planar counterparts [42].

4. Conclusions

We have successfully synthesized and characterized {octakis (alkylthio)phthalocyanato} nickel(II) and palladium(II) complexes. The length of the alkyl chain has considerable effects on ground and excited state symmetry and, by extention, the optical properties of the MPcs. The Pd(II) octakis(pentylthio)phthalocyanines showed a loss of symmetry due to dome-type distortion and the loss of symmetry its Ni(II) derivative was due to ring deformation. Symmetry was restored with longer alkyl chain length.

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