

Synthesis, characterization and spectroscopic investigation of azo-porphyrins

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Abstract

A series of novel covalently connected azonaphthalene porphyrin derivatives were prepared by linking azonaphthalene groups through a diazo-coupling reaction. They were characterized by UV–vis, IR, ^1H NMR and ESI-MS spectroscopic techniques. Azo–hydrazone tautomerism was studied using ^1H NMR and UV–vis techniques. The two chromophores of these derivatives exhibited their absorption spectroscopic properties and the effect of solvent upon the absorption ability was examined. In the fluorescence emission spectra, intermolecular fluorescence quenching was detected.

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Keywords: Porphyrin; Azonaphthalene; Diazo-coupling reaction; Keto–hydrazone form; UV–vis spectra; Fluorescence quenching

1. Introduction

Porphyrins and their derivatives have become the current focus of research due to their outstanding physico-chemical properties and applications in diverse fields. Well-designed porphyrin derivatives can act as models for natural photosynthetic system [1–4], and as molecular switches [5–7] and other organic photoelectric materials [8–10]. In these fields the investigation of photoinduced electron transfer is essential in understanding the mechanism and processes of these molecular scale electronic components.

Azo compounds are very important in the fields of dyes, pigments and advanced materials [11]. They can perform *cis–trans* isomerization under photochemical reactions and might undergo energy transfer [12,13]. Moreover, the azo–hydrazone tautomerism in disperse azo dyes [14–18] is one of the interesting research fields because it is very important for their optical stability and usage. It is explained by the proton transfer between the O and imine N atoms, which also has

potential use as the basis of optical data storage devices [19,20].

Recently, the compounds containing two chromophores, azo and porphyrin, including the azobenzene-linked porphyrins [21,22] and diporphyrins bridged by azo linkage [23–26], have been synthesized to develop new materials for molecular devices. But in these reported azo-porphyrin conjugates the azonaphthalene porphyrins did not involved. So the synthesis and their properties of the covalently linked two-component porphyrin system need further theoretical and experimental studies.

In the field of our investigation, we are interested in the development of a new class of azo-porphyrin chromophores. And very recently, we described the preparation and spectroscopic properties of the new azo-porphyrins that were synthesized using amino-porphyrins with phenol and naphthol [27]. As a continuation of our previous work in this area, we report herein the synthesis of the new azo-porphyrins **2** and **3**, in which the azonaphthalene groups are in the *para* position of the tetraphenylporphyrin and they are di-(**a**), tri-(**b**) and tetra-(**c**) substituted. The photochemical behaviors of the new compounds were examined using UV–vis absorption and fluorescence spectra. Since azo groups and porphyrins

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absorb light in different regions of the UV–vis spectrum, the two moieties should address independently. Moreover, new compounds **2** and **3** should confer molecular recognition properties on the system having hydrogen-bonding sites.

2. Experimental

2.1. General

All solvents and compounds were analytical grade reagents and were purified before use by distillation. Silica gel (100–200 mesh) was used for column chromatography. Melting points are uncorrected. ^1H NMR spectra were recorded in CDCl_3 using TMS as an internal standard on a Bruker AC-P300 at 300 MHz. Absorption spectra were obtained with Helios γ instrument. The IR spectra were recorded with a BIO-RAD FTS3000 FT-IR spectrometer. The mass spectra (MS) were recorded with a Finnigan LCQ mass spectrometer (ESI). The fluorescence spectra were obtained using Fluoro-Max-P spectrometer.

2.2. Synthesis

Amido-porphyrins (**1**) were synthesized as described by a previously reported method [28].

2.2.1. Synthesis of azo-porphyrins (**2**)

Porphyrins **1a–c** (1 mmol) were suspended in concentrated hydrochloric acid (1.6 mL) and distilled water (10 mL) in an ice bath. Cold sodium nitrite (0.21 g, 3 mmol) was added portion-wise and the reaction mixture was further stirred for 25 min at 0–5 °C. Residual HNO_2 was destroyed by adding carbamide and the reaction mixture was further stirred for 15 min to give a solution. The resultant diazonium solution after filtering was used in the coupling reaction. A mixture of the coupling component phenol or β -naphthol and 10% sodium carbonate was stirred as to get a clear solution. The diazonium mixture was added at –5 to 0 °C and the solution was stirred for 40 min before raising the pH to 8–9 with aqueous sodium carbonate. The resulting precipitate was filtered off and washed with a minimal amount of water. The crude product was chromatographed through a silica gel column (100–200 mesh) using petroleum ether, dichloromethane and ethyl acetate as the eluent. A violet band and purple crystalline solid porphyrins **2a–c** were obtained.

2.2.1.1. 5,15-Bis(4-nitrophenyl)-10,20-bis(4-(2-hydroxy-azonaphthyl)-phenyl) porphyrin (2a). $R_f = 0.51$ (petroleum ether/dichloromethane/ethyl acetate = 3:30:1, v/v/v); purple solid; 0.84 g, yield: 80%; mp > 300 °C. ^1H NMR (CDCl_3 , 300 MHz) δ : –2.73 (s, 2H, NH), 6.94 (d, $J = 9.9$ Hz, 2H, H*), 7.46 (m, 2H, ArH), 7.62–7.65 (m, 4H, ArH), 7.79–7.82 (m, 2H, ArH), 8.15–8.18 (m, 4H, ArH), 8.33–8.44 (m, 8H, ArH), 8.62–8.70 (m, 6H, ArH), 8.80 (d, $J = 3.9$ Hz, 4H, pyrrole-H), 9.01 (d, $J = 3.3$ Hz, 4H, pyrrole-H), 16.60 (s, 2H, OH); IR (KBr) ν/cm^{-1} : 3410, 3059, 2959, 2925, 2854, 1726, 1629, 1599, 1512, 1467, 1385, 1283, 1209, 1137, 1075, 963,

846, 799, 746; ESI-MS m/z found: 1045.7, calcd for $\text{C}_{64}\text{H}_{40}\text{N}_{10}\text{O}_6$: 1045.3 ($\text{M} + \text{H}$) $^+$.

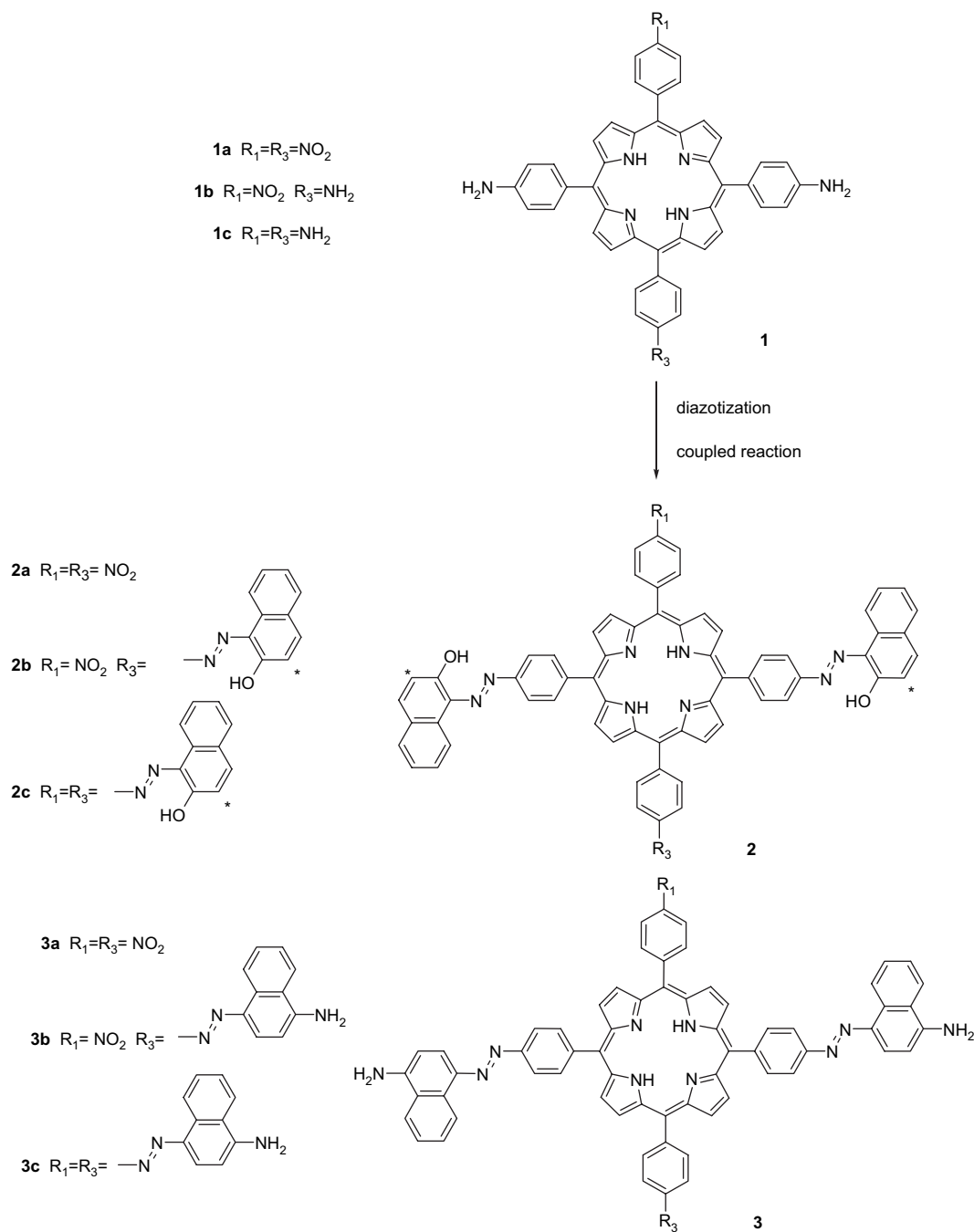
2.2.1.2. 5-(4-Nitrophenyl)-10,15,20-tris(4-(2-hydroxy-azonaphthyl)-phenyl) porphyrin (2b). $R_f = 0.22$ (petroleum ether/dichloromethane/ethyl acetate = 3:30:1, v/v/v); purple solid; 0.92 g, yield: 79%; mp > 300 °C. ^1H NMR (CDCl_3 , 300 MHz) δ : –2.69 (s, 2H, NH), 6.94 (d, $J = 9.3$ Hz, 3H, H*), 7.45–7.48 (m, 3H, ArH), 7.60–7.67 (m, 6H, ArH), 7.79–7.82 (m, 3H, ArH), 8.15–8.18 (m, 6H, ArH), 8.34–8.45 (m, 8H, ArH), 8.66–8.73 (m, 5H, ArH), 8.80–8.81 (m, 2H, pyrrole-H), 9.00 (s, 6H, pyrrole-H), 16.60 (s, 3H, OH); IR (KBr) ν/cm^{-1} : 3438, 3319, 2956, 2926, 2857, 1721, 1620, 1596, 1504, 1451, 1399, 1345, 1254, 1207, 1148, 965, 840, 798, 750; ESI-MS m/z found: 1171.8, calcd for $\text{C}_{74}\text{H}_{47}\text{N}_{11}\text{O}_5$: 1171.4 ($\text{M} + 2\text{H}$) $^+$.

2.2.1.3. 5,10,15, 20-Tetra(4-(2-hydroxy-azonaphthyl)-phenyl) porphyrin (2c). $R_f = 0.10$ (petroleum ether/dichloromethane/ethyl acetate = 3:30:1, v/v/v); purple solid; 1.06 g, yield: 82%; mp > 300 °C. ^1H NMR (CDCl_3 , 300 MHz) δ : –2.69 (s, 2H, NH), 6.94 (d, $J = 9.6$ Hz, 4H, H*), 7.45–7.48 (m, 4H, ArH), 7.60–7.67 (m, 8H, ArH), 7.79–7.82 (m, 4H, ArH), 8.15–8.18 (m, 8H, ArH), 8.34–8.45 (m, 8H, ArH), 8.66–8.73 (m, 4H, ArH), 8.99 (s, 8H, pyrrole-H), 16.60 (s, 4H, OH); IR (KBr) ν/cm^{-1} : 3426, 3316, 3075, 2925, 2854, 1720, 1620, 1599, 1556, 1502, 1453, 1400, 1346, 1256, 1149, 985, 966, 799, 753; ESI-MS m/z found: 1296.0, calcd for $\text{C}_{84}\text{H}_{54}\text{N}_{12}\text{O}_4$: 1296.4 ($\text{M} + 2\text{H}$) $^+$.

2.2.2. Synthesis of azo-porphyrins (**3**)

Porphyrins **1a–c** (1 mmol) were suspended in concentrated hydrochloric acid (1.6 mL) and distilled water (10 mL) in an ice bath. Cold sodium nitrite (0.21 g, 3 mmol) was added portion-wise and the reaction mixture was further stirred for 25 min at 0–5 °C. Residual HNO_2 was destroyed by adding carbamide and the reaction mixture was further stirred for 15 min to give a solution. The resultant diazonium solution after filtering was used in the coupling reaction. A mixture of the coupling component α -naphthylamine and 10% sodium acetate was stirred as to get a clear solution. The diazonium mixture was added at –5 to 0 °C and the solution was stirred for 40 min before dilution or raising the pH to 6–7 with aqueous sodium acetate. The resulting precipitate was filtered off and washed with a minimal amount of water. The crude product was chromatographed through a silica gel column (100–200 mesh) using petroleum ether, dichloromethane and ethyl acetate as the eluent. A violet band and purple crystalline solid porphyrins **3a–c** were obtained.

2.2.2.1. 5,15-Bis(4-nitrophenyl)-10,20-bis(4-(4-amino-azonaphthyl)-phenyl) porphyrin (3a). $R_f = 0.46$ (petroleum ether/dichloromethane/ethyl acetate = 3:30:1, v/v/v); purple solid; 0.77 g, yield: 74%; mp > 300 °C. ^1H NMR (CDCl_3 , 300 MHz) δ : –2.73 (s, 2H, NH), 4.73 (s, 4H, NH_2), 6.92–8.46 (m, 26H, ArH), 8.66 (d, $J = 8.1$ Hz, 2H, pyrrole-H), 8.80 (d, $J = 4.5$ Hz, 2H, pyrrole-H), 9.02 (s, 4H, pyrrole-H), 9.23



Scheme 1. Azo-porphyrin derivatives.

(d, 2H, $J = 8.4$ Hz, ArH); IR (KBr) ν/cm^{-1} : 3477, 3380, 3330, 2956, 2923, 2852, 1718, 1618, 1577, 1514, 1463, 1398, 1334, 1261, 1190, 1138, 964, 798; ESI-MS m/z found: 1043.7, calcd for $\text{C}_{64}\text{H}_{42}\text{N}_{12}\text{O}_4$: 1043.3 ($\text{M} + \text{H}$) $^+$.

2.2.2.2. 5-(4-Nitrophenyl)-10,15,20-tris(4-(4-amino-azonaphthyl)-phenyl) porphyrin (3b). $R_f = 0.28$ (petroleum ether/dichloromethane/ethyl acetate = 3:30:1, v/v/v); purple solid; 0.84 g, yield: 72%; mp > 300 °C. ^1H NMR (CDCl_3 , 300 MHz) δ : -2.67 (s, 2H, NH), 4.71 (s, 6H, NH_2), 6.92–8.46 (m, 32H, ArH), 8.66 (d, $J = 8.7$ Hz, 1H, pyrrole-H), 8.80

(d, $J = 5.1$ Hz, 1H, pyrrole-H), 9.03 (s, 6H, pyrrole-H), 9.23 (d, 2H, $J = 8.7$ Hz, ArH); IR (KBr) ν/cm^{-1} : 3456, 3392, 2954, 2922, 2850, 1618, 1572, 1514, 1460, 1398, 1332, 1259, 1223, 1192, 1138, 964, 796, 756; ESI-MS: m/z found 1167.7, calcd for $\text{C}_{74}\text{H}_{50}\text{N}_{14}\text{O}_2$: 1167.4 ($\text{M} + \text{H}$) $^+$.

2.2.2.3. 5,10,15, 20-Tetra(4-(4-amino-azonaphthyl)-phenyl) porphyrin (3c). $R_f = 0.12$ (petroleum ether/dichloromethane/ethyl acetate = 3:30:1, v/v/v); purple solid; 0.93 g, yield: 72%; mp > 300 °C. ^1H NMR (CDCl_3 , 300 MHz) δ : -2.62 (s, 2H, NH), 4.71(s, 8H, NH_2), 6.92–8.42 (m, 34H, ArH), 9.02

(s, 8H, pyrrole-H), 9.24 (d, 2H, $J = 8.1$ Hz, ArH); IR (KBr) ν/cm^{-1} : 3483, 3398, 2920, 2850, 1624, 1572, 1518, 1466, 1400, 1332, 1261, 1192, 1140, 966, 798, 758; ESI-MS m/z found: 1291.8, calcd for $\text{C}_{84}\text{H}_{58}\text{N}_{16}$: 1291.5 ($\text{M} + \text{H}$) $^{+}$.

3. Results and discussion

3.1. Synthesis and characterizations

The bi-(**a**), tri-(**b**) and tetra-(**c**) substituted azo-porphyrin conjugates **2a–c** and **3a–c** were prepared by diazotization of amino-porphyrins **1a–c** using concentrated hydrochloric acid at room temperature, and coupling with naphthol and naphthylamine (Scheme 1). The starting compounds **1a–c** were easily synthesized by deoxidization from tetranitrophenylporphyrin [27].

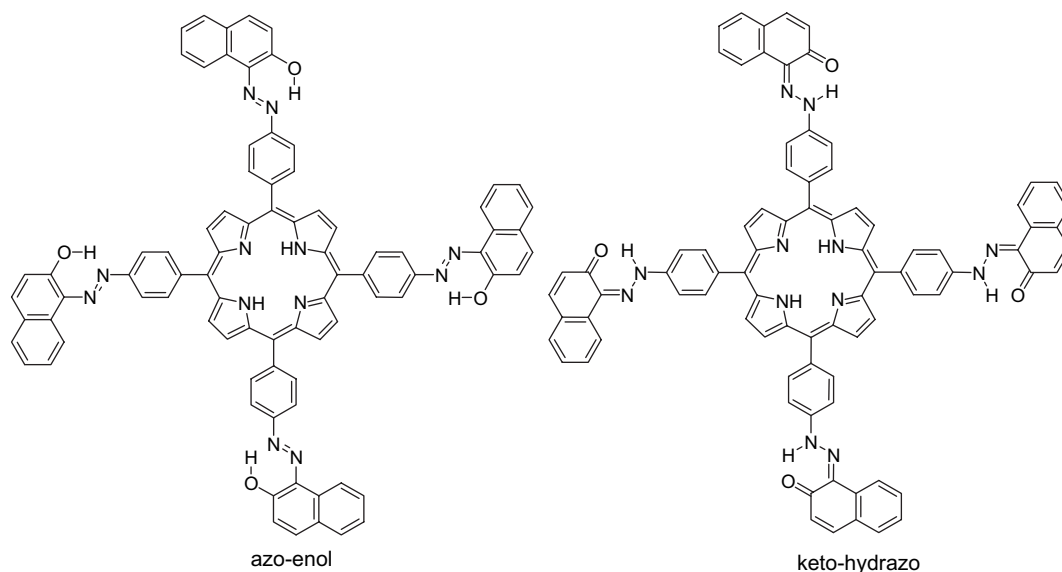
All reactions proceeded smoothly producing the corresponding azo compounds in good yields. The azo-porphyrin dyes were characterized by FT-IR, ^1H NMR, ESI-MS and UV–vis spectroscopic techniques.

The IR spectra of chromophores (**2a–c** and **3a–c**) showed a weak band within the range $3483\text{--}3395\text{ cm}^{-1}$ corresponding to ν_{OH} (compounds **2**) and ν_{NH} (compounds **3**). The low value revealed that the $-\text{OH}$ and $-\text{NH}$ groups were involved in intramolecular H-bonding. The IR spectra also showed a weak band or shoulder located at $2983\text{--}2854\text{ cm}^{-1}$ which was assigned to aromatic C–H, stretching vibration of the $\text{N}=\text{N}$ group leading to the band located in the $1467\text{--}1452\text{ cm}^{-1}$ region. It can be suggested from broad $-\text{OH}$ bands at $3440\text{--}3395\text{ cm}^{-1}$ in the infrared spectra that compounds **2** may exist as the azo–enol form in the solid state.

From the ^1H NMR spectra in the CDCl_3 , the NH protons of the porphyrins **2a–c** and **3a–c** appeared as one singlet around $\delta -2.7$. Also, the ^1H NMR spectra of compounds

2a–c and **3a–c** showed single or double bands at about $\delta 9.0$ corresponding to β -pyrrolic protons. But due to the presence of nitro-group, four and two β -pyrrolic protons shifted to around $\delta 8.8$ in compounds **2a** and **2b**, respectively, while the β -pyrrolic protons shifted to around $\delta 8.6$ and 8.8 in compounds **3a** and **3b**, respectively. Furthermore, the pyrrolic protons showed doublets in compounds **2a** and **3a**. This indicated that compounds **2a** and **3a** should be *trans* but not *cis* substituted. This was identical with Vicente's results [29]. The OH protons of compounds **2** appeared as one singlet at $\delta 16.6$ in the ^1H NMR spectra, while NH_2 of compounds **3** appeared at $\delta 4.7$. In addition, H^* proton in compounds **2** shifted to high fields (around $\delta 6.9$) and the coupling constant was up to $J = 9.3\text{--}9.9$. These findings suggest that compounds **2** exist as the keto–hydrazo form in CDCl_3 (Scheme 2) according to literature results [30,31].

UV–vis absorption spectra were measured using a Helios γ spectrophotometer in the wavelength range $200\text{--}800\text{ nm}$. The absorption spectra of porphyrins showed the typical Soret and Q-bands at the concentration of about $5 \times 10^{-6}\text{ mol/L}$. From the UV–vis absorption spectra (Fig. 1) the porphyrinic Soret bands are broader compared with that of TPP (*meso*-tetraphenylporphyrin), indicating a ground state interaction of the azonaphthalene groups and porphyrin chromophore. Moreover, the Soret bands had strong tailings on the red edge of the absorption bands in compounds **3**. This may result from $n \rightarrow \pi^*$ absorption of the *Z*-azonaphthalene moiety. This was identical with Hombrecher's results [24]. The absorption bands of compounds **2** and **3** show relatively weak and broadened bands in the azo groups' $\pi \rightarrow \pi^*$ region at $309\text{--}356\text{ nm}$. And the azo groups' bands of compounds **2** were markedly blue-shifted compared with those of compounds **3**. The Q-bands of new compounds **2** and **3** showed insignificantly affected; however, the azonaphthalene group



Scheme 2. The tautomeric forms of compound **2c**.

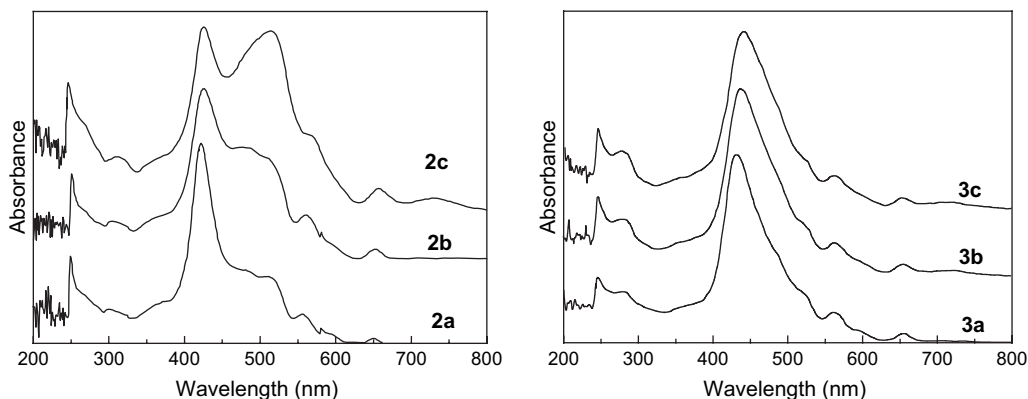


Fig. 1. UV-vis spectra of compounds **2** and **3** in chloroform (5×10^{-6} mol/L).

peaks near 510 nm of compounds **2** cover-up a Q-band of porphyrin moiety near 517 nm. It is clear furthermore that these peaks of **2** follow an increasing trend in height according to the increase of azonaphthalene group in **a**, **b** and **c**. Such findings also indicated that compounds **2** may exist as the keto–hydrazo form in chloroform [32].

As demonstrated in Fig. 2 the fluorescence peak intensity of **2** decreases significantly in the order of di-(**a**), tri-(**b**) and tetra-(**c**) azo-group substituted compounds. The porphyrin fluorescence of **2b** and **2c** are quenched by 7% and 45%, respectively, as compared to compound **2a**. And it is clear that the emission near 720 nm reduces markedly. These results evidently indicate that substantial amount of electron transfer occurs from the azonaphthalene group to the porphyrin chromophore in the excited state. In addition, compounds **2** have more emission peaks at 851 nm compared with TPP when excited at the azophenyl group with absorption maximum at 356 nm. In contrast, the fluorescence peak intensity increases with the addition of the azonaphthalene substituent. However, the fluorescence peaks of compounds **3** are not significantly quenched except the peak around 610 nm. Thus covalent connection of the chromophores **3** does not greatly perturb the excited state properties of the porphyrin component.

3.2. Solvent effect

The absorption spectra of azo dyes **2a–c** and **3a–c** were recorded in various solvents as a concentration of about 5×10^{-6} mol/L; the results are summarized in Table 1. And Fig. 3 shows the absorption spectra of compound **3b** in various solvents. The visible absorption spectra of compounds **2** and **3** were found to show that the typical Soret and Q-bands of porphyrin bands and azo bands did not significantly change except those in acetic acid. As is apparent in Fig. 3, the Soret band red-shifted to around 566 nm and the broad Q-band at 772 nm were observed.

4. Conclusions

In conclusion, the diazo-coupling reactions are shown in Scheme 1. This pathway is the most convenient to give the best yield of diazo-coupling porphyrin compounds. Six new bi-, tri- and tetra-substituted azonaphthalene-porphyrin conjugates with hydrogen-bonding groups immersed had been synthesized. The characterization of these new compounds had been described and the ^1H NMR and absorption spectra results of dyes **2** revealed that these compounds do exist in forming azo–enol form and keto–hydrazo form species.

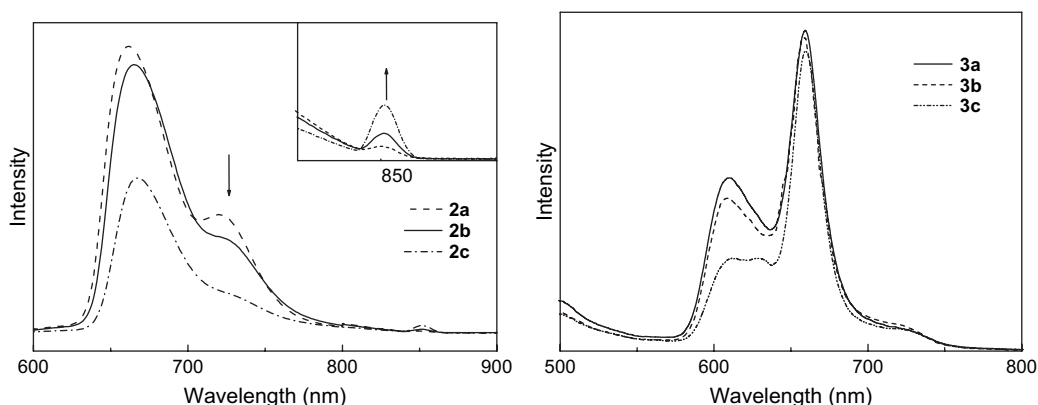


Fig. 2. The fluorescence spectra of porphyrins **2** and **3** in chloroform (5×10^{-6} mol/L).

Table 1
Influence of solvent on λ_{max} (nm) of dyes **2a–c** and **3a–c**

No.	Chloroform			Ethyl acetate			Acetonitrile			DMF			Acetic acid		
	λ_{azo}	λ_{Soret}	λ_{Q}	λ_{azo}	λ_{Soret}	λ_{Q}	λ_{azo}	λ_{Soret}	λ_{Q}	λ_{azo}	λ_{Soret}	λ_{Q}	λ_{azo}	λ_{Soret}	λ_{Q}
2a	303	424	502	321	415	550	326	414	580	311	425	559	318	440	695
	512		559 580 653	511		647	515		651	512		591 652	536		
2b	305	426	489	321	419	554	330	420	580	312	427	561	316	441	705
	511		559 580 653	512		653	512		650	508		594 652	534		
2c	309	426	513	323	423	551	321	422	580	309	426	565	316	438	705
	514		566 656 725	511		650	512		650	508		654	515		
3a	356	432	520	352	425	558	353	417	553	356	424	500	365	450	776
			564 592 652			598 652			593 650			561 653	567		
3b	356	436	523	353	441	561	351	420	555	356	424	501	366	561	772
			562 652			599 654			593 651			561 653			
3c	354	440	520	352	449	560	357	448	561	353	421	500	366	533	773
			562 658			601 654			589 652			563 654			

The electronic spectra of new compounds exhibited the typical bands of azo, Soret and Q, and the intramolecular electronic communications between azonaphthalene and porphyrin units were detected. However, they did not significantly change along with the various solvents. Owing to fluorescence quenching, chromophores **2** showed intramolecular electron transfer. Thus, they are potentially capable of molecular sensing or switching applications. More detailed studies of the photochemistry of these compounds are presently under investigation.

Acknowledgements

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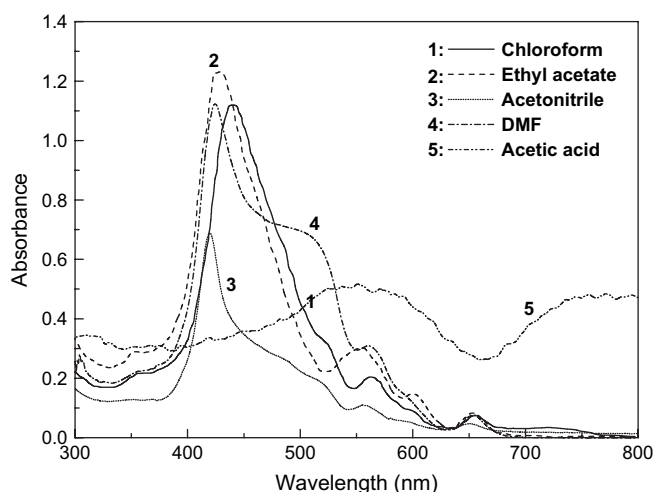


Fig. 3. Absorption spectra of dye **3b** in various solvents.

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