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Short communication

Synthesis of 3,5-diaryl substituted indole derivatives and its selective iodide ion chemosensing

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ABSTRACT

In this contribution, we have synthesized series of 1-ethyl-3,5-bis[2-(aryl)ethenyl]-1H-indole based derivatives with different functional groups using Wadsworth–Emmons coupling. The sensing ability of the receptors has been studied for halides like tetrabutylammonium fluoride, chloride, bromide, iodide and bisulphate by UV-vis spectroscopy methods. In THF solution, compound **A–E** exhibits excellent selectivity with iodide ions over the other ions like tetrabutylammonium bromide (TBABr), tetrabutylammonium chloride (TBACl), tetrabutylammonium fluoride (TBAF) and tetrabutylammonium bisulphate (TBAHSO₄). The indole cores were observed effectively and selectively recognized biologically important iodide was reported. Significant absorption change was observed only for tetrabutylammonium iodide and no change with other anions. The absorption spectra indicated the formation of complex between host and guest is in 1:1 stoichiometric ratio. The association constants of **A–E** for iodide ions were found to be $3.2 \times 10^4 \, \text{M}^{-1}$, $3.9 \times 10^4 \, \text{M}^{-1}$, $4.2 \times 10^3 \, \text{M}^{-1}$, $2.9 \times 10^3 \, \text{M}^{-1}$ and $2.14 \times 10^3 \, \text{M}^{-1}$, respectively.

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1. Introduction

Molecular recognition of anionic species plays an essential role in a large number of chemical, biological and environmental processes [1–3]. The search of a chemosensor for recognition and sensing of specific anionic analytes through naked eye, electrochemical and optical responses are emerging research areas of considerable importance [4]. The practical recognition of anions has continued to be a more challenging issue than the recognition of cations, which has been fully developed for nearly forty years [5]. The variety of geometry shapes of anions and synthetic difficulty in building selective anionic receptive sites make the design and synthesis of anion sensor intellectually more demanding and practically less predictable [6]. Among various important anionic analytes, the iodide ion is one of the most significant, since play an essential role in biological activities, such as thyroid function and neurological activity. In many systems the iodide content of milk and urine is often required for metabolic, nutritional, and epidemiological studies of thyroid disorder [7]. In addition

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** Corresponding author. Tel.: +65 65196593; fax: +65 84317476. *E-mail addresses:* doctorindira@gmail.com (V.K. Indirapriyadharshini), mperajen@nus.edu.sg (R. Murugan). the elemental iodine has been frequently used in many areas of chemistry for synthesizing valuable molecules/intermediates such as drugs, dyes and molecular electronics. There are few reports on the fluorescent recognition of iodide using small molecules and conjugated polymers [8]. In most cases, hydrogen bonding between the N-H of the molecule and anions was used for the recognition, without the involvement of hydrogen bonds, quaternized ammonium hosts [9], azamacrocycles [10], and phosphonium hosts [11] are known to form anion complexes. A cyclic peptide has been reported for the anion sensing includes iodides, bromides, chlorides and fluorides, but there is no selectivity [12]. Bis-imidazolinium [13], adenine [14], trifluoroacetyl aminophthalimide based derivatives [15], benzimidazole based tripodal receptor [16], pyrenyl-appended triazole-based calix[4]arene [17], silver nanoparticles [18], N-fused tetraphenylporphyrin [19], 4,7diaryl indole-based derivatives [20] and macrocyclic binuclear copper (II) complex [21] are reported for the selective iodide sensors. The present receptors are designed based on quaternized ammonium hosts bonds in the receptor aligned in parallel; it may effectively make a complex with iodide ions. Herein we report the synthesis of new indole based sensors containing conjugated moieties and their selective sensing of iodide ions. The electronic nature of its substituent was used to tune the selectivity and the photophysical properties of the chemosensors.

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2. Experimental

2.1. Materials and methods

All reagents were purchased from Aldrich, Fluka and/or Merck and were used without further purification unless otherwise stated. All reactions were carried out with dry, freshly distilled solvents under anhydrous conditions or in an inert atmosphere. THF was purified by distillation from sodium in the presence of benzophenone under nitrogen atmosphere. The NMR spectra were collected on a Bruker ACF 400 spectrometer with DMSO-d₆ as solvent. Elemental analyses were done in VarioMicro analyzer. UV-vis spectra were recorded on a PG Instrument Ltd. spectrophotometer. Compounds N-ethyl indole [22] and Nethyl indole-5-carboxyaldehyde [23] were prepared according to the published procedures. Phosphonates A_1-D_1 diethyl benzylphosphonate [24], diethyl (quinolin-8-ylmethyl) phosphonate [25], diethyl (3-methoxybenzyl) phosphonate [26], diethyl (3fluorobenzyl) phosphonate [27] required for stilbene synthesis were prepared by Arbusov reaction between the appropriate aromatic methyl bromide and trietylphosphite. Tetrahydrofuran of HPLC grade was purchased from Aldrich and used throughout the experiments as solvent for chemosensor studies. All the titration measurements were carried at room temperature. The THF solutions of anions were prepared from their tetrbutylammonium salts of analytical grade and used for titration. The anion binding behaviors of 3,5-diaryl substituted indole derivatives were determined by UV-vis spectroscopic studies.

2.2. General experimental procedure for the synthesis of the A–E

To diethyl phenyl methyl phosphonate **A**₁ (0.471 g, 2.06 mmol) in dry THF (10 mL) was added NaH (0.043 g, 1.72 mmol). After initial effervescence the suspension was stirred for 1 h at 85 °C under nitrogen. To this 1-ethyl indole-3,5-carboxyaldehyde **6** (0.100 g, 0.689 mmol) in dry THF (10 mL) was added drop wise and the mixture heated to reflex at 85 °C for 12 h. Then cooled to room temperature and poured in to water and extracted with ethylacetate (2 × 20 mL). The organic layer was added brine solution (2× 10 mL) and layer was separated and dried with Na₂SO₄, filtered and concentrated. The crude product was purified by column chromatography using hexane:ethylacetate (60:40) as eluent.

2.2.1. 1-Ethyl-3-[(E)-2-phenylethenyl]-1H-indole (A)

Brown solid; 80 mg, 47%; ¹H NMR (400 MHz, DMSO–d₆): δ 1.38 (t, *J* = 3.32 Hz, 3H), 4.23 (q, *J* = 7.20 Hz, 2H), 7.06–7.11 (m, 4H), 7.33–7.44 (m, 3H), 7.52 (d, *J* = 8.04 Hz, 1H), 7.58 (d, *J* = 7.84 Hz, 2H), 7.72 (s, 1H), 8.02 (d, *J* = 7.88 Hz, 1H); ¹³C NMR (100 MHz, DMSO–d₆): δ 138.92, 136.83, 129.05, 128.63, 126.70, 126.20, 125.90, 123.83, 122.40, 122.25, 120.51, 120.24, 113.45, 110.61, 15.77; *m/z* 248.32 (M⁺). Anal. Calcd. for C₁₈H₁₇N: C, 87.41; H, 6.93; N, 5.66%. Found: C, 87.37; H, 6.90; N, 5.78%.

2.2.2. 1-Ethyl-3,5-bis[(E)-2-phenylethenyl]-1H-indole (B)

Brown solid; 25 mg, 44.4%; ¹H NMR (400 MHz, DMSO-d₆): δ 1.41 (t, *J* = 7.24 Hz, 3H), 4.22 (q, *J* = 7.20 Hz, 2H), 7.13–7.18 (m, 5H), 7.23–7.29 (m, 5H), 7.39 (q, *J* = 7.64 Hz, 2H), 7.47–7.64 (m, 4H), 7.73 (s, 1H), 8.22 (s, 1H); ¹³C NMR (100 MHz, DMSO-d₆): δ 138.94, 138.21, 136.61, 130.44, 129.67, 129.14, 129.06, 128.97, 127.43, 126.79, 126.64, 126.55, 126.06, 126.01, 124.19, 122.18, 120.78, 119.54, 113.99, 110.97, 15.82; *m/z* 350.46 (M⁺). Anal. Calcd. for C₂₆H₂₃N: C, 89.36; H, 6.63; N 4.01%. Found: C, 89.39; H, 6.70; N, 4.06%.

2.2.3. 1-Ethyl-3,5-bis[(E)-2-(quinolin-8-yl)]-1H-indole(C)

Yellow solid; 100 mg, 44%; ¹H NMR (400 MHz, DMSO–d₆): δ 1.44 (t, *J* = 7.12 Hz, 3H), 4.28 (q, *J* = 7.12 Hz, 2H), 7.57–7.68 (m, 7H), 7.47 (s, 1H), 7.84–7.91 (m, 3H), 8.22–8.24 (m, 2H), 8.36–8.51 (m, 3H), 9.01 (s, 2H); ¹³C NMR (100 MHz, DMSO–d₆): δ 149.74, 149.58, 145.07, 144.86, 136.53, 136.30, 135.52, 131.64, 129.74, 128.68, 128.36, 128.33, 127.03, 126.68, 126.53, 126.20, 124.69, 123.75, 123.13, 121.64, 121.54, 121.30, 120.85, 119.45, 118.80, 114.36, 110.80, 15.48; *m*/z 452.34 (M⁺). Anal. Calcd. for C₃₂H₂₅N₃: C, 85.11; H, 5.58; N, 9.31%. Found: C, 85.08; H, 5.67; N, 9.38%.

2.2.4. 1-Ethyl-3,5-bis[(E)-2-(3-methoxyphenyl)ethenyl]-1H -indole (D)

Off brown solid; 40 mg, 49%; ¹H NMR (400 MHz, DMSO-d₆): δ 1.39 (t, *J*=7.20 Hz, 3H), 3.80 (s, 6H), 4.21 (q, *J*=7.24 Hz, 2H), 6.76–6.82 (m, 2H), 7.08 (s, 1H), 7.13 (s, 4H), 7.24–7.30 (m, 2H), 7.42–7.54 (m, 2H), 7.70 (s, 1H), 8.19 (s, 1H); ¹³C NMR (100 MHz, DMSO-d₆); δ 159.64, 159.62, 139.95, 139.20, 136.14, 130.23, 129.63, 129.54, 129.14, 128.62, 126.11, 125.53, 123.61, 122.04, 120.23, 119.25, 118.71, 113.46, 112.84, 111.08, 110.69, 110.54, 55.04, 15.36; *m*/*z* 410.23 (M⁺). Anal. Calcd. for C₂₈H₂₇NO₂: C, 82.12; H, 6.65; N, 3.42%. Found: C, 82.14; H, 6.71; N, 3.38%.

2.2.5. 1-Ethyl-3,5-bis[(E)-2-(3-fluorophenyl)ethenyl]-1H-indole (E)

Brown oil; 38 mg, 62%; ¹H NMR (400 MHz, DMSO–d₆): δ 1.38 (t, *J* = 7.16 Hz, 3H), 4.22 (q, *J* = 7.16 Hz, 2H), 6.99–7.06 (m, 2H), 7.10 (d, *J* = 15.92 Hz, 1H), 7.27 (d, *J* = 16.40 Hz, 1H), 7.35–7.55 (m, 10H), 8.24 (s, 1H); ¹³C NMR (100 MHz, DMSO–d₆): δ 163.97, 163.89, 161.56, 141.31, 141.23, 140.52, 140.44, 136.33, 131.51, 130.56, 130.47, 130.43, 130.34, 129.21, 128.94, 126.08, 124.41, 123.37, 122.42, 121.80, 120.58, 119.39, 113.64, 113.43, 113.25, 112.91, 112.69, 112.13, 111.92, 111.39, 110.62, 15.34; *m/z* 386.45 (M⁺). Anal. Calcd. for C₂₆H₂₁F₂N: C, 81.02; H, 5.49; N, 3.63%. Found: C, 81.06; H, 5.41; N, 3.65%.

3. Results and discussion

The synthetic route to the 1-ethyl-3,5-bis[2-(aryl)ethenyl]-1Hindole derivatives A-E is outlined in Scheme 1. Treatment of indole 1 with ethyl bromide in dry THF in the presence of sodium hydroxide afforded N-ethyl indole 2 which was Vilsmeier-Haack formylation with phosphorous oxychloride in DMF to the N-ethyl indole-5-carboxyaldehyde. The Wadsworth-Emmons condensation of N-ethyl indole-3-carboxyaldehyde was treated with corresponding triethylphosphite salts A_1-D_1 in the presence of 2.5 equiv of sodium hydride in THF, the reaction mixture was refluxed for 12 h which gave compound A. A similar sequence followed by preparation of compounds B-E. All the reactions are very straight forward. The alkyl chain in A-E led to a reasonable level of solubility in a variety of organic solvents such as CH₂Cl₂, CHCl₃, MeOH and THF. All the phosphonates A_1-D_1 required for stilbene synthesis were prepared by Arbusov reaction between the appropriate aromatic methyl bromide and trietylphosphite (Scheme 1). They were well characterized by MS, elemental and ¹H, ¹³C, NMR spectroscopic methods before using them in application. NMR spectroscopy is one of the principal techniques which give us the structural information about receptor molecules. NMR spectra were obtained using DMSO-d₆ as solvent. Compound E gave a multiple at around δ 6.99–7.06 ppm corresponding to the vinyl proton indicating the formation of conjugated and δ 7.35–7.55 ppm corresponding to the aromatic protons.



Scheme 1. Synthesis of 1-ethyl-3,5-bis[2-(aryl)ethenyl]-1H-indole derivatives A-E.



Fig. 1. Absorption spectra of A–E in acetronitrile and THF solution.

3.1. Photophysical study of 3,5-diaryl indoles A-E

The optical properties of **A–E** were investigated by UV–vis spectroscopy on THF solutions (10^{-5} M) at room temperature. Absorption spectra of these derivatives show two maxima (λ_{max}), one in the range 300–330 nm and another above 300 nm. All the receptor shows absorption band at (λ_{max}) 336, 339, 379, 306 and 310 nm in the absence of an anion. Since there is no significant difference in the conjugation length, there are no significant changes in the λ_{max} with changes in the functional group. The

fluorescence spectra of the **A–E** were recorded with the excitation wavelength corresponding to their maximum absorption wavelength. The titration experiments of **A–E** performed by a UV–vis spectroscopic method using tetrabutylammonium halides and bisulphate at room temperature. Fig. 1 shows the absorption spectra of compounds **A–E** in different solvents. The calculated HOMO–LUMO energy gaps are in good agreement with the experimental values obtained from the UV–vis spectra (Table 1). All the new compounds were characterized using various analytical techniques.

Table 1	
Photophysical properties of 1-ethyl-3,5-bis	[2-(aryl)ethenyl]-1H-indoles A-E.

Compound	Absorption λ_{max}				Emission λ_{em}	λ_{onset}	Band gap ^a (eV)	$\varepsilon_{\rm max}$
	THF	MeoH	CH ₃ CN	Toluene				
А	336	331	332	337	415	356	3.48	5.3×10^5
В	339	300	301	306	410	381	3.25	$1.1 imes 10^5$
С	379	376	368	312	413	417	2.97	$1.8 imes 10^5$
D	306	303	308	278	408	346	3.58	$1.3 imes 10^5$
E	310	307	302	281	423	350	3.54	$1.2 imes 10^5$

^a Calculated from onset wavelength.



Fig. 2. (a) Absorption spectra of **E** in THF after addition of various tetrabutylammonium salts in 1 M solution. The concentration of **E** was 2.67×10^{-5} M. (b) Change in the absorption spectra of **E** at different concentration of tetrabutylammonium iodide in THF solution: (i) 0 μ M, (ii) 10 μ M, (iii) 30 μ M, and (iv) 60 μ M.



Fig. 3. (a) Change in the absorption spectra of **A** at different concentration of tetrabutylammonium chloride in THF solution: (i) 10 μ M, (ii) 30 μ M, and (iii) 60 μ M. (b) Change in the absorption spectra of **E** at different concentration of tetrabutylammonium fluoride in THF solution: (i) 10 μ M, (ii) 30 μ M, and (iii) 60 μ M.

3.2. UV-vis titration study of A-E with anions

The variation of indole through the introduction of UV–vis active aryl groups at 3, 5 position is expected to improve the photophysical properties for the chemosensing studies. Considering the biological, environmental analytical relevance of anions, such as F⁻, Cl⁻, Br⁻ and I⁻ the sensing properties of **A–E** were investigated using the corresponding tetrabutylammonium salts in THF solution. Interestingly, these **A–E** detect only iodide among other common anions such as fluorides, chlorides, bromides and bisulphate. In the case of addition of fluoride, chloride, bromide and bisulphate ions into the **A–E** solution, no significant changes in absorption (see supporting data) however the addition of the iodide ion results in a blue shift in the absorption spectra (Fig. 2a and b). The absorption changes of **A** with a solution of tetrabutylammonium chloride and **E** with solution of tetrabutylammonium fluoride were shown in Fig. 3.

3.3. Job's plot

The association constants k_a of **A–E** for iodide were calculated on the basis of Benesi–Hildebrand plot [28] and were found to be $3.2 \times 10^4 \text{ M}^{-1}$, $3.9 \times 10^4 \text{ M}^{-1}$, $4.2 \times 10^3 \text{ M}^{-1}$, $2.9 \times 10^3 \text{ M}^{-1}$ and $2.14 \times 10^3 \text{ M}^{-1}$, respectively. The stoichiometry of the complex formed was determined by Job's plot [29] and turned out to be 1:1 stoichiometry in Fig. 4. This indicated that sensors had reached a saturation level as it binds to its anions guest. The nature of other anions, such as tetrabutylammonium halides (F⁻, Cl⁻, Br⁻ and HSO₄⁻) does not influence the absorption changes. The

changes observed on the absorption spectra of **E** (60μ M in THF) upon titrating with a solution of tetrabutylammonium solution like iodide, bromide, chloride, fluoride and bisulphate. The change was observed only with iodide not other anions like fluoride, bromide, chloride and bisulphate (Fig. 2a).

It is interesting to note that the absorption spectra of **A–E** can be modulated by addition of iodide ion. The quenching effects of the all



Fig. 4. Job's plots for complexation of E with iodide determined by UV-vis spectroscopy in THF.



Fig. 5. Relative UV–vis absorption responses of **E** in the presence of tetrabutylammonium iodide at various concentrations in THF.

Table 2

Absorption responses of **A–E** upon addition of iodides.

Compounds	$\lambda_{max} \left(nm \right)$	$\Delta \lambda_{max}$	
	I ⁻ free ^a	Presence of I ^{- b}	
Α	336	295	-41
В	339	295	-44
С	379	289	-90
D	306	293	-13
E	310	291	-19

^a Absorption in THF.

^b Addition of iodides.

the derivatives absorption in the presence of tetrabutylammonium iodide salts have been investigated in detail (see supporting data). Comparative UV–vis absorption responses of **E** in the presence of tetrabutylammonium iodide at various concentrations are shown in Fig. 5. The addition of iodide salts to **A–E** produced a significant shift in the absorption spectra and the results are summarized in Table 2.

4. Conclusions

In summary, we have synthesized and structurally characterized indole based chemosensors, as an ideal receptor for iodide. The receptor binds iodide strongly, showing good selectivity for it over other halides. The new receptors are capable of discriminating anions that can be monitored from their λ_{max} and absorption intensity.

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.saa.2011.10.067.

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