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Colorimetric detection of aliphatic primary amines and a molecular logic gate based on a photochromic phenoxyquinone derivative

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ABSTRACT

A photochromic phenoxyquinone-based, sensor for aliphatic primary amine was developed. An efficient trans-to-ana photoisomerization was occurred when 6-(4'-methoxyphenoxy)-5,12naphthacenequinone in acetonitrile was irradiated with 365 nm UV light. The photochemically generated "ana" quinone intermediate was found to undergo nucleophilic substitution reactions by aliphatic primary amines to afford a large bathochromic shift in the absorption spectra, allowing colorimetric detection of the aliphatic primary amines. Neither aromatic nor secondary and tertiary amines were able to promote the bathochromic spectral shift of the phenoxyquinone. Removal of the phenoxy group by a primary amine was confirmed by using ¹H NMR analysis and a preparative scale reaction. In addition, colorimetric detection of the aliphatic primary amine was demonstrated to be feasible with phenoxyquinone-containing PDMS films. Since the large bathochromic shift occurs only in the presence of UV light and aliphatic primary amine, the sensor system functions a molecular AND logic gate.

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1. Introduction

Photochromic materials experience reversible structural changes that interconvert two distinctly colored isomeric forms upon irradiation at specific wavelength [1–4]. Owing to their light driven molecular switching properties, photochromic compounds have been incorporated into many interesting systems, including molecular switches [5–8], optical memory devices [9–11], holographic gratings [12,13], logic devices [14–16], and drug delivery vesicles [17]. Azobenzene, spiropyran, phenoxyquinone, and bisthienylethene derivatives are representative photochromic compounds. These molecules undergo various types of structural isomerization reactions, such as cis-trans isomerization (azobenzene), ring opening (spiropyran), phenyl group migration (phenoxyquinone), or ring closure (bisthienylethene), upon UV irradiation. Moreover, the reverse processes are readily promoted by irradiation with visible light.

Recently, applications of photochromic materials in the chemosensor area have received great attention [18–21]. That molecular sensing properties can be triggered by light is a very intriguing phenomenon since it enables "On-Off" switching of sensory functions to be governed by UV or visible light irradiation.

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1010-6030/\$ - see front matter © 2012 Elsevier B.V. All rights reserved. http://dx.doi.org/10.1016/j.jphotochem.2012.04.003 Shiraish et al. demonstrated that a photochromic spiropyran could be utilized to detect cyanide anion in a sensitive and reversible fashion [18]. The photochemically generated merocyanine (MC) form of spiropyran was readily reacted with cyanide anion to yield an adduct. The formation of spiropyran-cyanide adduct resulted in the colorimetric change of the solution. More recently, they reported a coumarin-spiropyran conjugate sensor system for the detection of cyanide anion [19]. Unlike the simple spiropyran derivative, the fused coumarin-spiropyran system allowed a fluorometric detection of cyanide anion. Thus, no fluorescence was observed with a photochemically generated ring-opened coumarin fused spiropyran molecule. A strong fluorescence was monitored when the fused sensor molecule binds with cyanide anion. We also reported a cyanide-specific chemosensor based on a polythiophene having spiropyran moieties as pendent groups [20]. The fluorescence emission of polythiophene was quenched by fluorescence resonance energy transfer (FRET) from the polymer backbone to MC form of the spiropyran when the spiropyran-polythiophene conjugate was irradiated with UV light. Recovery of the fluorescence was observed when the solution was exposed to cyanide anion, allowing a FRET-based cyanide sensor. Most recently, we reported a cyanide selective photochromic chemosensor system based on a phenoxyquinone derivative [21]. We observed that addition of cyanide anion to a UV irradiated solution of a phenoxynaphthacene quinone derivative brought about a significant change in the absorption spectra that enabled detection of cyanide ion in a highly selective and sensitive manner. A carbanion intermediate was shown to be responsible for the long wavelength absorption band that is generated by cyanide addition.

The development of reliable detection methods for volatile aliphatic amines is important in the fields of environmental monitoring, food quality control, and chemical device processing. Due to their relative ease of handling and monitoring, colorimetric and fluorometric chemosensors have been extensively studied for the detection of the volatile aliphatic amines [22–29]. However, no photochromic, colorimetric amine sensors have been described to date. As part of our continuing interest in the design of photochromism-based chemosensors [19,20], we have carried out an investigation that has led to the development of a facile colorimetric method for the sensitive detection and differentiation of aliphatic primary amines from their secondary and tertiary counterparts. In addition, the feasibility of a molecular logic gate with the photochromic amine sensor system is described.

2. Experimental

2.1. Apparatus

¹H NMR and ¹³C NMR spectra were recorded on a Varian Unitylnova (300 MHz) spectrometer, using CDCl₃ as solvent. Melting points were determined on an IA9100X1 (Thermo Scientific Barnstead Electrothermal). IR spectra were recorded on a MAGNa-IR E.S.P (Thermo Fisher Scientific, Inc). UV–vis spectra were examined on an Agilent 8453 spectrophotometer.

2.2. Materials

Primary, secondary and tertiary amines investigated in this study were purchased from Sigma–Aldrich. SYLGARD[®] 184 silicon elastomer base and SYLGARD[®] 184 silicon elastomer curing agent were purchased from DOW CORNING. 6-Chloro-5,12-naphthacenequinone was prepared according to the reported method [29].

2.3. Synthesis

2.3.1. Synthesis of

6-(4'-methoxyphenoxy)-5,12-naphthacenequinone (1)

The known compound, 6-(4'-methoxyphenoxy)-5,12naphthacenequinone (1) was prepared according to the reported general method [30]. A mixture of 6-chloro-5,12naphthacenequinone (150 mg, 0.51 mmol), 4-methoxyphenol (76 mg, 0.62 mmol) and potassium carbonate (92 mg, 0.67 mmol) in dry DMF (10 mL) was heated at 110 °C for 12 h. The mixture was poured into acidic water (500 mL). The precipitate was collected, washed with water and dried. Recrystallization from ethyl acetate led to pure phenoxyquinone **1**. Yield: 90 mg (46%). m.p.: 201 °C; ¹H NMR (300 MHz, CDCl₃): δ = 8.86 (1H, s), 8.37–8.13 (4H, m), 7.80-7.63 (4H, m), 6.82 (4H, s), 3.75 (3H, s); ¹³C NMR (75 MHz, in $CDCl_3$): $\delta = 182.9$, 181.1, 154.5, 153.4, 152.9, 136.0, 135.4, 134.3, 133.6, 133.5, 131.9, 130.7, 130.3, 130.1, 129.8, 127.6, 127.0, 126.9, 125.0, 121.0, 115.5, 114.8, 55.6.

2.3.2. Synthesis of 6-(butylamino)tetracene-5,11-dione (4)

A solution of 6-(4'-methoxyphenoxy)-5,12naphthacenequinone (1) (40 mg, 0.105 mmol) in acetonitrile (105 mL) was irradiated with 365 nm UV light (1 mW/cm²) for 24 h. n-Butylamine (0.21 mL, 2.10 mmol) was added to the solution and the resulting solution was stirred for 2 h at room temperature. Evaporation of the solvent and unreacted n-butylamine gave a reddish residue. The residue was subjected to a silica gel column chromatography (ethylacetate/hexane (v/v, 2:1)) to afford the n-butylamine adduct **4** (20 mg, 58%). m.p.: 158–159 °C; ¹H NMR (300 MHz, in CDCl₃): δ = 8.55 (1H, m), 8.46 (1H, m), 8.14 (1H, m), 7.99 (1H, m), 7.82 (1H, m), 7.73 (2H, m), 7.59 (2H, m), 4.12 (2H, q, *J* = 8 Hz), 1.99 (2H, q, *J* = 8 Hz), 1.63 (2H, m), 1.04 (3H, t, *J* = 8 Hz); ¹³C NMR (75 MHz, in CDCl₃): δ = 183.8, 177.5, 160.2, 136.1, 135.7, 134.1, 133.4, 133.0, 131.5, 131.2, 130.8, 129.6, 129.4, 128.9, 128.8, 126.7, 120.0, 106.3, 50.2, 33.6, 20.9, 14.4; IR (KBr) ν max (cm⁻¹): 3432, 3303, 3056, 2956, 2927, 2869, 1974, 1945, 1834, 1724 1654, 1583, 1552, 1527, 1425, 1375, 1328, 1247, 1083, 956, 892, 757, 698; HRMS *m/z* 329.1419 (calcd C₂₂H₁₉NO₂, 329.1416).

2.3.3. Synthesis of 6-hydroxynaphthacenequinone (7)

Method A (photochemical): A solution of 6-(4'methoxyphenoxy)-5,12-naphthacenequinone (1) (40 mg, 0.105 mmol) in acetonitrile (105 mL) was irradiated with 365 nm UV light (1 mW/cm²) for 24 h. Diethylamine (0.21 mL, 2.10 mmol) was added to the mixture and stirred for 4 h at room temperature. Evaporation of the solvent and unreacted diethylamine gave a residue which was washed with acetone/THF (1:1) solution to yield the hydroxyquinone **7** (18 mg, 52%). m.p.: 254–256 °C; ¹H NMR (300 MHz, in CDCl₃): δ = 8.54 (1H, d, *J* = 8 Hz), 8.40 (2H, m), 8.33 (1H, s), 8.01 (1H, d, *J* = 8 Hz), 7.83 (2H, m), 7.74 (2H, qd, *J* = 2.8 Hz).

Method B (chemical): A mixture of homophthalic anhydride (1.85 mmol) and NaH (60% in mineral oil, 1.94 mmol) in anhydrous THF (25 mL) was stirred at 0 °C for 3 min. A solution of 1,4-naphthoquinone (1.85 mmol) in anhydrous THF (5 mL) was added to the mixture. The mixture was stirred at 0 °C for 20 min and stirred for 11 h at ambient temperature. The reaction mixture was quenched with saturated aqueous NH₄Cl (20 mL) and then extracted with 1 M HCl solution and methylene chloride. The organic layer was washed with saturated aqueous NaCl, dried (MgSO₄), and concentrated under reduced pressure. The residue was washed with acetone to give the hydroxyquinone **7** (299 mg, 59%) [32].

2.4. PDMS film sensor

2.4.1. Preparation

A solution of phenoxyquinone **1** (10 mg) in chloroform (1 mL) was mixed with SYLGARD[®] 184 slicone elastomer base (20 g) and stirred vigorously until well mixed. SYLGARD[®] 184 curing agent (2 g) was added to the PDMS mixture. Bubbles in the PDMS mixture was removed by a vacuum chamber for 1 h. The degassed PDMS mixture was slowly poured into a square Petri dish (12.5 cm × 12.5 cm). The PDMS mixture was cured at room temperature for 2 days to give the phenoxyquinone embedded PDMS film (film thickness is 950 µm).

2.4.2. Sensor test

Liquid amine (2 mL) was placed in a 4 mL glass vial and the vial was cap-sealed. After generating saturated amine vapor by heating the vial at the boiling point of respective amines, the vial was open and a phenoxyquinone embedded PDMS film, which was irradiated with 365 nm UV light (1 mW/cm²) for 10 min, was placed on the top of the vial for 2 min and the color change of the film was monitored.

3. Results and discussion

In order to explore the feasibility of colorimetric aliphatic primary amine sensor, a photochromic 6-(4'-methoxyphenoxy)-5,12-naphthacenequinone (1) displayed in Scheme 1 was prepared according to the literature method [30]. The phenoxyquinone 1 is expected to undergo a trans-ana isomerization reaction that generates "ana"-quinone 2 (Scheme 1) [30]. The "ana"-quinone forms of phenoxyquinones are known to undergo nucleophilic substitution



Scheme 1. Photoinduced isomerization of 6-(4'-methoxyphenoxy)-5,12-naphthacenequinone (1) and formation of an amine-quinone adduct 3.

reactions with primary amines to generate amine-quinone adducts **3** [31]. In the context of colorimetric sensing, the substitution process is interesting since it should result in a bathochromic shift in the absorption spectrum owing to hydrogen bonding between the hydrogen on the amine group and the adjacent carbonyl group in adduct **3**. In addition, the presence of the electron donating amine group in **3** facilitates formation of a charge transfer excited state that results in a red shift of the absorption maximum. As a consequence of these phenomena, the photochemically generated "ana" quinone **2** can serve as a colorimetric chemosensor for primary amines.

In order to investigate the above proposal, an acetonitrile solution $(25 \,\mu\text{M})$ containing the phenoxyquinone **1** was irradiated for 30 min using a handheld laboratory 365 nm UV light lamp. Formation of the "ana" quinone form **2** was confirmed by observing characteristic UV-vis absorption peaks in the 400–500 nm region (Fig. 1).

To the photoirradiated solution was added various amines while monitoring the spectral changes. As shown by the spectra displayed in Fig. 2A, addition of primary aliphatic amines, such as n-propylamine, n-butylamine, n-hexylamine, and ndecylamine, to the UV irradiated phenoxyquinone 1 results in large bathochromic shifts of the absorption maximum. Importantly, secondary amines, such as piperidine and diethylamine do not induce the bathochromic shift. As expected, almost no changes in the absorption spectra are observed in the presence of tertiary amines. Interestingly, the aromatic primary amine aniline does not induce the red shift of the photochemically generated "ana" quinone form **2**. Thus, the spectral changes in quinone **2** are both specific for aliphatic primary amines and significantly large to be readily observed. It should be noted that a solution containing unirradiated phenoxyquinone 1 does not undergo any spectral changes when amines (primary, secondary and tertiary amines) are added



Fig. 1. UV-vis absorption spectra of an acetonitrile solution containing $25 \,\mu$ M of the phenoxyquinone **1** upon irradiation with 365 nm UV light ($1 \,mW/cm^2$) for 0 (a), 6 (b), 12 (c), 18 (d), and 30 (e) min.

(Fig. 2B). This observation demonstrates that the observed spectral change promoted by primary amines originates from a reaction of the "ana"-quinone form **2**.

The next phase of the current investigation focused in more detail on the colorimetric transition of the photoirradiated quinone 1 by the model primary amine n-butylamine. In Fig. 3A are shown UV–vis absorption spectra of a 365 nm UV irradiated solution of the phenoxyquinone 1 (25 μ M) in the presence of various concentrations of n-butylamine.

As expected, absorption at 523 nm appears and increases as the concentration of n-butylamine increases. The presence of an observed isosbestic point at 492 nm indicates that the aminequinone reaction produces a single product without the existence



Fig. 2. UV-vis absorption spectra of irradiated (A) and unirradiated (B) acetonitrile solutions (25μ M) of the phenoxyquinone 1 in the presence of various amines (20 molar equiv. to 1). The spectra were recorded $5 \min$ (A) and $30 \min$ (B) after addition of the amine.



Fig. 3. (A) UV-vis absorption spectra of a 365 nm UV-irradiated (30 min, 1 mW/cm²) acetonitrile solution (25 μ M) of the phenoxyquinone 1 in the presence of 0 (a), 5 (b), 10 (c), 15 (d), 20 (e), and 25 (f) μ M of n-butylamine. The inset shows vials containing photochemically generated "ana" quinone 2 in the absence and presence of n-butylamine. (B) Plot of absorbance at 523 nm as a function of n-butylamine concentration. The inset contains a plot of the absorption ratio vs. n-butylamine concentration in the lower micromolar range.

of intermediates. Based on the plots of absorbance at 523 nm as a function of butylamine concentration shown in Fig. 3B, the detection limit of butylamine was determined to be $1.2 \,\mu$ M. Since the saturation of product formation was observed at a much higher concentration of butylamine ($125 \,\mu$ M) than the quinone used ($25 \,\mu$ M), a possible equilibrium between the initially formed product and butylamine may exist.

In order to gain more information about the nature of the interaction between n-butylamine and photogenerated "ana"-quinone 2, ¹H NMR spectroscopic analysis was carried out (Fig. 4). The four protons on the phenoxy ring of quinone **1** appear as a singlet at 6.82 ppm due to the presence of similarly electron donating para substituents (Fig. 4A). This singlet, however, is transformed to an AB quartet upon 365 nm UV irradiation of the solution (Fig. 4B). In addition, a downfield shift of the aromatic phenyl proton resonances occurs upon irradiation, indicating that the electron withdrawing quinone moiety is produced. Comparison of the aromatic phenoxy protons before and after UV irradiation suggests that the "trans" and "ana" quinone forms exist as a 1:1.3 mixture after 365 nm UV irradiation. Addition of n-butylamine to this solution results in the complete disappearance of the "ana" quinone form and simultaneous appearance of new resonances at 6.76 ppm which are associated with 4-methoxyphenol (Fig. 4C and E). Thus, the results of the ¹H NMR analysis demonstrate clearly that 4-methoxyphenol



Fig. 4. ¹H NMR (300 MHz) spectra of a CDCl₃ solution containing 2 mM of phenoxyquinone **1** before (A), after 365 nm UV irradiation for 4 h (B), after addition of 2 equiv. of n-butylamine to the UV irradiated solution (C). ¹H NMR spectra of independently prepared n-butylamine adduct 4 (D) and commercial 4-methoxyphenol (E).



Fig. 5. UV-vis absorption spectra of a 365 nm UV-irradiated acetonitrile solution $(25 \,\mu\text{M})$ of phenoxyquinone **1** in the presence of n-butylamine (solid) and n-butylamine adduct **4** isolated from a preparative scale reaction (dash).

is liberated from the "ana" quinone form **2** when n-butylamine is added. To demonstrate that the displacement reaction promoted by n-butylamine does indeed take place, the n-butylamine adduct **4** (Scheme 2) was generated by using a preparative scale reaction and analyzed using ¹H NMR spectroscopy (Fig. 4D). Comparison of the spectra shown in Fig. 4C and D indicates that n-butylamine adduct **4** formation is indeed responsible for the UV-vis and ¹H NMR spectroscopic changes observed in the experiments described above (Fig. 5).

Interestingly, addition of secondary amines, such as diethylamine and piperidine, to solutions of the photoirradiated quinone **1** results in hypsochromic shifts of the absorption maximum (Fig. 2A). We initially thought that amine–quinone adducts (e.g. **5**, Scheme 2), were being produced in these processes. However, the results of preparative scale reactions show that the only product generated in these processes is 6-hydroxynaphthacenequinone **7** (Scheme 2). It is believed that the initially formed secondary amine adducts (e.g. **5**) are highly unstable in that they undergo rapid hydrolysis by adventitious water present in the solution to produce the stable hydroxyquinone **7** via intermediate **6**.



Scheme 2. Reaction pathways to the generation of the amine adduct 4 and the hydroxyquinone 7.



Fig. 6. ¹H NMR (300 MHz) spectra of a CDCl₃ solution containing 2 mM of phenoxyquinone 1 after addition of 2 equiv. of diethylamine to the UV irradiated solution (A). ¹H NMR spectra of 6-hydroxynaphthacenequinone 7 isolated from a photochemical reaction (B) and from an independent chemical synthesis (C).

In order to prove the hydroxyquinone 7 was the sole product from the secondary amine-photogenerated quinine reaction, ¹H NMR spectroscopic analysis was conducted. As displayed in Fig. 6A, addition of 2 equiv. of diethylamine to the photochemically generated ana quinone 2 resulted in the formation of hydroxyquinone 7. The presence of the hydroxyquinone 7 was confirmed by isolation from the reaction mixture (Fig. 6B) and by independent chemical synthesis (Fig. 6C) (see Section 2 for details) [32].

Stimulated by the interesting feature of the sensor material **1**, we took a further step to test the selectivity of the quinone **1** as a colorimetric chemosensor for primary amine in the presence of secondary amine. Thus, the response of the photochemically generated "ana" quinone **1** to butylamine was evaluated in the presence of various concentrations of diethylamine. As displayed in Fig. 7, nearly the same final absorbance at 523 nm was observed (Fig. 7, c–h), indicating that no remarkable interference of the secondary amine was monitored.

The ability to selectively colorimetric detect primary aliphatic amines and not their secondary and tertiary counterparts is not limited to the solution phase. In Fig. 8 are shown PDMS films containing phenoxyquinone **1** after exposure to saturated vapors of various amines. As expected, the unirradiated PDMS films do not undergo color changes when any amine is present (Fig. 8A). In addition, irradiation using 365 nm UV light of a phenoxyquinone **1** containing film for 10 min leads to generation of a yellow color (Fig. 8B, ctrl), which is indicative of the occurrence of the



Fig. 7. UV-vis absorption spectra of a phenoxyquinone **1** solution (CH₃CN, 25 μ M) before (a) and after (b) 365 nm UV irradiation for 30 min. UV-vis spectra (c-h) of a UV irradiated quinone solution in the presence of various molar ratios of butylamine and diethylamine (250:0 (c), 250:50 (d), 250:100 (e), 250:150 (f), 250:200 (g), and 250:250 (h) μ M).



Fig. 8. Photographs of unirradiated (A) and 365 nm UV irradiated (B) PDMS films containing phenoxyquinone 1 after exposure to saturated vapors of n-butylamine, diethylamine and triethylamine for 2 min.

photoisomerization reaction. Exposure of the yellow film produced in this way to n-butylamine vapor induces a color change to pale purple (Fig. 8B), showing that the amine adduct was generated.

Since the generation of absorbance at 523 nm is only possible when the quinone **1** is converted to ana form **2** by UV irradiation and the quinone **2** reacts with primary amine, construction of a

Table 1

Truth table for the two-input logic gate.

Input 1 (365 nm UV)	Input 2 (n-butylamine)	Output (abs at 523 nm)
0	0	0
1	0	0
0	1	0
1	1	1

two-input AND logic gate is possible [33]. As presented in the truth table (Table 1), no output signal (absorbance at 523 nm) is generated when input 1 (365 nm UV irradiation) and input 2 (primary aliphatic amine) are absent. In addition, the output signal is not monitored when only one input signal is present. Thus, the output signal is observed only when the two input signals are present, thus allowing a molecular AND logic gate.

4. Conclusions

In summary, the study described above has led to the development of a photochromic phenoxyquinone **1** based, colorimetric chemosensor for the detection of aliphatic primary amines. Nucleophilic displacement of the phenoxy group in the photochemically generated "ana" quinone form 2 by an aliphatic primary amine promotes a large bathochromic shift of the absorption maximum that enables colorimetric detection of the aliphatic primary amines. Removal of the phenoxy group by a primary amine was confirmed by using ¹H NMR analysis and a preparative scale reaction. In addition, colorimetric detection of the aliphatic primary amine was demonstrated to be feasible with phenoxyquinone-containing PDMS films. Finally, we proved that the photochromic sensor system could act as a molecular AND logic gate in response to two input signals (UV light and aliphatic primary amine). Although numerous colorimetric chemosensors have been reported, those based on photochromism probe molecules are rare. Thus, the strategy described above should serve as an important addition to the chemosensor area.

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