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Detection of Triethylamine on Supramolecular 3-[(E)-(4-acetylphenyl)diazenyl]-4-hydroxybenzaldehyde Compound

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Abstract. Triethylamine (TEA) has been recognized as one of the sources for irritations that occurred in the system of respiratory, ocular, and dermal tissues. Therefore, the development of materials that able to detect the TEA is still highly required. In this work, the synthesis of a novel supramolecular azo compound for potential detection of TEA in solution, which was 3-[(E)-(4-acetylphenyl)diazenyl]-4-hydroxybenzaldehyde was reported. The successful formation of the azo compound was supported by the thin layer chromatography (TLC), Fourier transform infrared (FTIR), and ultraviolet-visible (UV-vis) spectroscopies. The TLC result showed different retention factor (R_f) values between the synthesized compound and its reactants, while the FTIR and the UV-vis spectra revealed the presence of C–N, C=O, C=N, and N=N groups that confirmed the formation of the azo compound. The detection of the TEA was examined by monitoring the changes of the azo compound absorbance before and after the addition of TEA. The higher concentration of the added TEA led to the higher absorbance at 467 nm. The linear plot between absorbance and TEA concentration indicated the potential use of the supramolecular azo compound to detect the TEA in solution.

INTRODUCTION

Triethylamine (TEA) is widely used in various chemical industries, especially as a base catalyst for several organic reactions [1,2]. When the amount is high, TEA could give glaucopsia hazards and interfere with the respiratory system as well as ocular and dermal tissues [3,4]. Due to its moderate toxicity and flammable properties, the detection of TEA as gas and dissolved compound in our aquatic environment is still required. While the quantitative detection of TEA has been developed by chromatography analysis [5,6], fast detection of TEA is still required. Recent works have reported the development of various solid materials as gas sensors for the detection of TEA [7-10]. However, there is less attention to the development of sensing materials for the detection of TEA in aqueous solution, which is also an important process to reduce the hazards of the TEA.

In this work, the 3-[(E)-(4-acetylphenyl)diazenyl]-4-hydroxybenzaldehyde was synthesized as a potential material to detect TEA in an aqueous solution. Azo compounds are attractive materials as they could be synthesized from a diazonium salt and a coupling component [11,12] with a reasonable yield and reaction time. Owing to their unique properties, several azo compounds have been reported to exhibit good selectivity to detect cations [13,14], anions [15,16], as well as a neutral compound [17]. A lawsone-based azo dye was developed to give a response for Cu^{2+} and Fe^{3+} [13], while the rhodanine-based azo dye was reported to act as a spectrophotometric sensor for Fe^{3+} [14]. On the other hand, azo dye chemosensor based on solvent yellow 4 was reported to give good selectivity for the detection of fluoride ions [15] and the coumarin based azo dyes were reported for the detection of fluoride and acetate ions [16]. Another azo dye having a boronic acid group was reported for the detection of sugar [17]. All these studies showed that we still could explore new azo compounds and their applications for various analytes. In the present study, a novel azo compound was synthesized for the detection of TEA from diazotization of 4-

aminoacetophenone and 4-hydroxybenzaldehyde in the presence of potassium carbonate (K_2CO_3) as the base source. It was demonstrated that the 3-[(E)-(4-acetylphenyl)diazenyl]-4-hydroxybenzaldehyde as a supramolecular azo compound could be used as a chemosensor for the detection of TEA.

EXPERIMENTAL SECTION

General

The chemicals used in this study were 4-aminoacetophenone ($C_6H_4NH_2COCH_3$, Sigma-Aldrich, 99%), potassium carbonate (K_2CO_3 , Aldrich, 99.995%), sodium nitrite ($NaNO_2$, Merck), hydrochloric acid (Merck, 37%), 4-hydroxybenzaldehyde (C_6H_4OHCHO , Sigma-Aldrich, 98%), sodium hydroxide (NaOH), and triethylamine ($(CH_2CH_3)_3N$, Merck). No purification step was involved before the use of these chemicals. Thin layer chromatography (TLC), Fourier transform infrared (FTIR, JASCO FTIR-6800) and ultraviolet-visible (UV-vis, JASCO V-760) spectrophotometers were used to characterize the azo compound product.

Procedure

Synthesis of Azo Compound

Potassium carbonate (0.771 g), 4-aminoacetophenone (1.564 g, 0.012 mol), and distilled water (31.25 mL) were mixed and stirred at 50 °C until they were dissolved completely. The solution was cooled to room temperature before it was kept under the temperature of 0–5 °C. The sodium nitrite (0.647 g, 0.009 mol) was then added to the solution, while stirring under the temperature of 0–5 °C, following the dropwise addition of hydrochloric acid (3.125 mL). The solution was kept under the same temperature for 15 minutes and labeled as solution A. In another erlenmeyer, a solution of sodium hydroxide (10%, 6 mL) was prepared and 4-hydroxybenzaldehyde (1.698 g, 0.014 mol) was added into the solution, denoted as solution B, which was also cooled at temperature of 0–5 °C. Solution A was then added dropwise to solution B, which was kept stirred at the temperature. The reaction was allowed to occur for 4 hours to form a solid product having a brown color. The obtained product was recrystallized by water to separate the azo compound from the chalcone by-product and was consequently dried in an oven at 110 °C overnight. The collected product was 1.452 g, giving a product yield of 58%.

Characterizations of Azo Compound

The azo compound product and the reactants (4-aminoacetophenone and 4-hydroxybenzaldehyde) were tested by TLC, where the TLC plate was put inside a beaker and saturated with the mixture of ethyl acetate and hexane (1:1). The TLC plate was dried and the plate was irradiated by ultraviolet (UV) light of 254 nm to visualize the spots from the product and the reactants. The azo compound product was also characterized by FTIR spectroscopy using the attenuated total reflection (ATR) method at room temperature. The measurement was carried out in the range of 400–4000 cm^{-1} . The absorption of the azo compound product in the UV and the visible region was also measured by UV-vis spectroscopy in the range of 200–600 nm. For such purpose, a certain concentration of the azo compound (0.3 mM, 3 mL) was prepared by dissolving the azo compound in the ethanol and water (2:3) as the solvent.

Detection of TEA over Azo Compound

The detection of TEA was carried out by a UV-vis spectrophotometer. The azo compound solution (0.3 mM, 3 mL) was prepared similarly as mentioned above and measured as the initial absorbance without the addition of the TEA. Meanwhile, the TEA solution (200 mg/L) was prepared as the stock solution, which was then dissolved in the azo compound solution to various concentrations of 2, 4, 6, and 8 ppm. As example, to prepare 3 mL of TEA (2 mg/L) in azo compound solution (0.3 mM), 90 μ L of azo compound solution (1 mM) and 30 μ L of TEA (200 mg/L) were added to 2.88 mL of ethanol-water solvent (2:3). The mixture was mixed completely and kept at room temperature about 1 hour before each measurement. The UV-vis spectra were recorded in the range of 200–600 nm and the changes before and after the addition of the TEA were monitored at 467 nm.

RESULTS AND DISCUSSION

Synthesis and Characterizations of Azo Compound

As shown in Figure 1, the reaction between diazonium salt and the coupling agent, in this case 4-hydroxybenzaldehyde, would produce the azo compound [11,12]. The diazotization reaction would occur between 4-aminoacetophenone and sodium nitrite in the presence of potassium carbonate to produce diazonium salt, which was then reacted with 4-hydroxybenzaldehyde in the presence of sodium hydroxide to produce the 3-[(E)-(4-acetylphenyl)diazenyl]-4-hydroxybenzaldehyde. The visualized spots of 4-hydroxybenzaldehyde, 4-aminoacetophenone, and the azo compound are shown in Figure 2. It was clear that the three compounds gave different spot positions, which resulted in the different retention factor (R_f) values. R_f values have been generally used to identify different organic compounds. The R_f values for 4-hydroxybenzaldehyde, 4-aminoacetophenone, and the azo compound were determined to be 0.7, 0.8, and 0.9, respectively, which indicated that the product was successfully obtained as it showed a different R_f value from those of the reactants.

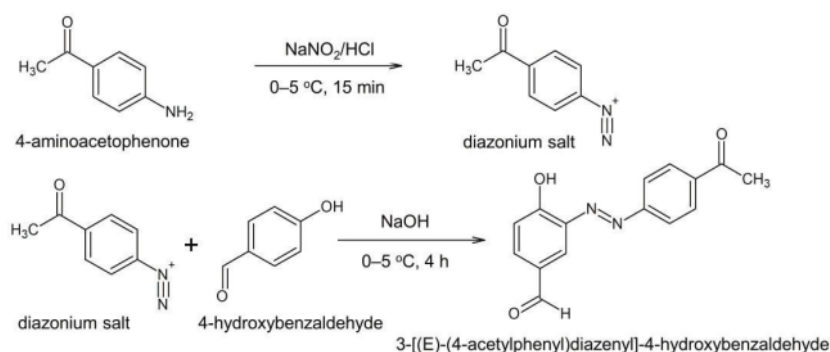


FIGURE 1. Synthesis reactions to obtain the 3-[(E)-(4-acetylphenyl)diazenyl]-4-hydroxybenzaldehyde

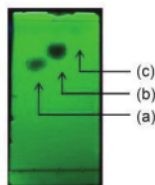
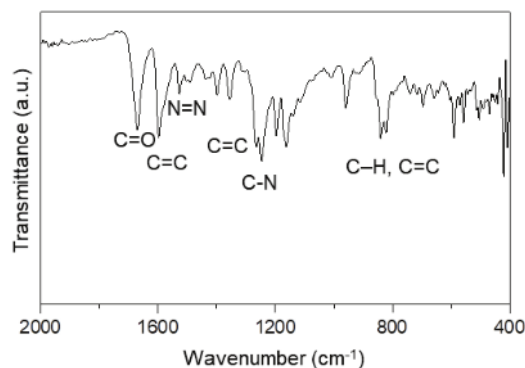


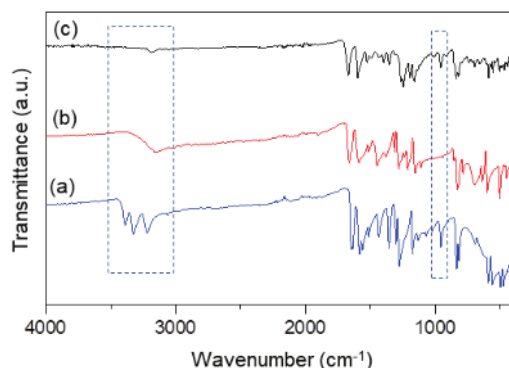
FIGURE 2. Visualized TLC spots of (a) 4-aminoacetophenone, (b) 4-hydroxybenzaldehyde, and (c) 3-[(E)-(4-acetylphenyl)diazenyl]-4-hydroxybenzaldehyde

23
In order to further confirm the successful formation of the azo compound, the FTIR spectrum was measured and shown in Figure 3. The important functional groups in the azo compound related to the azo chromophore ($\text{N}=\text{N}$), $\text{C}-\text{N}$ stretching (aryl), and $\text{C}=\text{C}$ stretching (aryl) in the absorption range of $1200-1600\text{ cm}^{-1}$ [18]. The evidence of the formation of the azo compound could be observed at 1526 and 1498 cm^{-1} assigned as $\text{N}=\text{N}$ groups. The presence of $\text{C}-\text{N}$ stretching (aryl) could be observed at $1240-1280\text{ cm}^{-1}$, while the $\text{C}=\text{C}$ stretching could be observed at around 1600 and 1400 cm^{-1} . In addition, the strong absorption of $\text{C}=\text{O}$ stretching could be observed in the 1676 cm^{-1} as well as the $\text{C}-\text{H}$ and $\text{C}-\text{C}$ bendings at $700-1000\text{ cm}^{-1}$. All these vibration peaks revealed that the important functional groups in the 3-[(E)-(4-acetylphenyl)diazenyl]-4-hydroxybenzaldehyde could be observed and identified, suggesting the successful formation of the azo compound. Besides, as depicted in Figure 4, the azo compound showed a different FTIR spectrum from those of the reactants. Figure 4 (a) shows that 4-aminoacetophenone gave a medium $\text{N}-\text{H}$ stretching in the range of $3200-3300\text{ cm}^{-1}$, which was not observed in the azo product. As for the 4-

hydroxybenzaldehyde, the C–H bending from alkyl group at 957 cm^{-1} was absent as shown in Figure 4 (b). These differences highlighted the formation of azo compound.



7
FIGURE 3. FTIR spectrum of 3-[(E)-(4-acetylphenyl)diazenyl]-4-hydroxybenzaldehyde



17
7
FIGURE 4. FTIR spectra of (a) 4-aminoacetophenone, (b) 4-hydroxybenzaldehyde, and (c) 3-[(E)-(4-acetylphenyl)diazenyl]-4-hydroxybenzaldehyde

The optical property of the 3-[(E)-(4-acetylphenyl)diazenyl]-4-hydroxybenzaldehyde was examined by UV-vis spectroscopy as depicted in Figure 5. As could be observed, the azo compound absorbed light from UV to visible region up to 600 nm, in good agreement with its light brown color as shown as an inset in Figure 5. The azo compound has a strong absorption peak below 200 nm, several peaks at 237, 266, 337, and 377 nm, as well as a shoulder peak around 470 nm. The peak below 200 nm could correspond to the presence of $\pi \rightarrow \pi^*$ from the alkyl group [19]. On the other hand, the peaks at 237, 266, 337, and 377 nm could be assigned to the electron transitions at the Ar C–N [20], C=O [19,20], N=N [19,20], and C=N groups, respectively. Besides, the shoulder peak at 470 nm showed the $n \rightarrow \pi^*$ absorption as the character of the azobenzene-like compounds [19]. Therefore, this UV-vis spectrum supported the formation of the azo compound.

To investigate the capability of the 3-[(E)-(4-acetylphenyl)diazenyl]-4-hydroxybenzaldehyde compound to detect TEA in solution, various concentrations of TEA (2–8 ppm) were prepared and added to the azo compound solution. Their UV-vis spectra were measured and shown in Figure 6 (a)–(e). The TEA solution was colorless, and after it was added into the azo compound solution, the solution turned its color from light brown to dark brown color. This interesting color change phenomena could be observed as an inset in Figure 6 that showed the representative example when TEA solution (2 ppm) was added into the azo compound. As shown in Figure 6 (a)–(e), the absorbance of the azo compound decreased with the addition of TEA, except for the visible peak at around 467 nm. Since TEA did not have absorption in this visible region and this absorption peak was slightly increased

with the increase of the TEA concentration, there were certain interactions between azo compound and TEA. The most likely occurred interactions would be electrostatic interactions, even solid materials functionalized by amines have been reported to act as good adsorbents for the removal of azo dyes [21,22]. It has been generally proven that the intramolecular electrostatic interactions could be one of the reasons to give such change in the absorption properties [23].

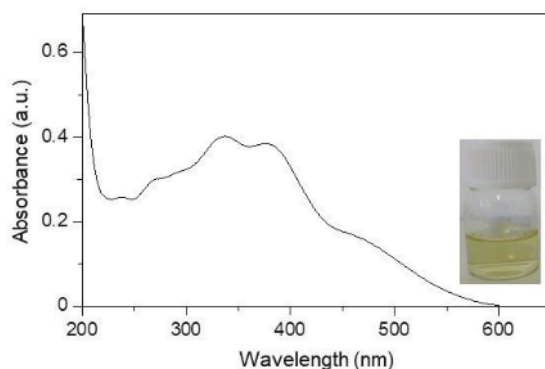


FIGURE 5. UV-vis spectrum of 3-[(E)-(4-acetylphenyl)diazenyl]-4-hydroxybenzaldehyde and its respective color appearance as shown as an inset.

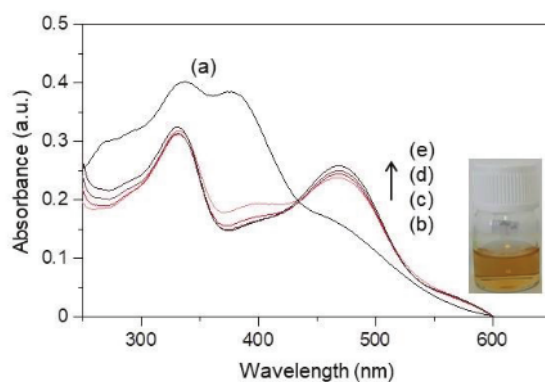


FIGURE 6. UV-vis spectra of 3-[(E)-(4-acetylphenyl)diazenyl]-4-hydroxybenzaldehyde (a) in the absence and presence of TEA solution having concentrations of (b) 2, (c) 4, (d) 6, and (e) 8 ppm. The solution (b) is also shown as an inset.

The interactions were investigated further to see if they could be used for the detection of the TEA. Figure 7 shows the plot between the absorbance at 467 nm and the concentration of TEA. The obtained plot was linear with a very high correlation coefficient, which suggested that the 3-[(E)-(4-acetylphenyl)diazenyl]-4-hydroxybenzaldehyde could be potentially used to detect the TEA compounds at the current concentration range. While the absorbance changes were not obvious due to the low concentration of TEA, this study demonstrated the possible use of the supramolecular azo compound as the chemosensor. Further investigations are still required to measure a larger concentration range and develop approaches to increase efficiency.

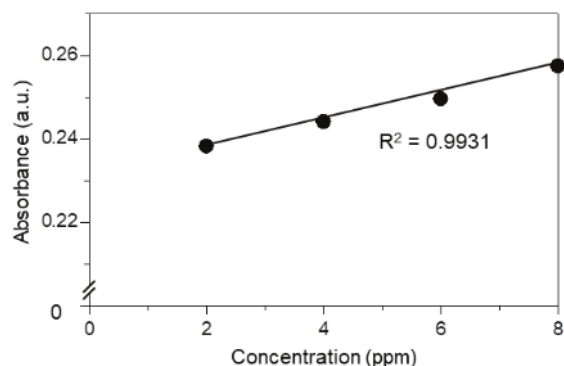


FIGURE 7. A linear plot between the absorbance at 467 nm and the TEA concentration

CONCLUSIONS

The 3-[(E)-(4-acetylphenyl)diazenyl]-4-hydroxybenzaldehyde compound was successfully prepared by a diazotization reaction between 4-aminoacetophenone and sodium nitrite, followed by the coupling reaction with 4-hydroxybenzaldehyde, and characterized by TLC, FTIR, and UV-vis spectrophotometers. The azo compound well interacted with the TEA by giving an absorption band in visible region around 467 nm. The absorbance increased with the increase of the TEA concentrations and the linear correlation plot was obtained. This result showed the potential use of the 3-[(E)-(4-acetylphenyl)diazenyl]-4-hydroxybenzaldehyde compound to detect TEA in solution.

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