



## Synthesis and Characterization of Some New Azo-Schiff Bases as Energy Rich Candidate Compounds Derived from 1, 5-Diaminoanthraquinone by Fusion Method

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### Abstract

This work, which can be considered as a complement to our previous studies concerning chemical energy storage systems developing programmed, proposes the synthesis of new azo-Schiff bases derived from 1, 5-diaminoanthraquinone (DAAQ) resulting in an improvement in their absorption spectra. Five new derivatives (An1-An5) were synthesized by gentle fusing of (1 mmol of DAAQ) with (2 mmol) of early prepared azo derivatives of 2-hydroxy benzaldehyde. Medium to good yield was recorded on applying this method. Microwave irradiation of the same mixtures led to decomposition of the starting azo materials whereas prolonged reflux in DMSO was unproductive.

**Keywords:** 1, 5-Diaminoanthraquinone (DAAQ), storage systems, microwave, fusion, supercapacitor electrodes

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## تحضير وتشخيص مركبات أزو- قواعد شف جديدة كمواد غنية بالطاقة واعدة

### مشتقة من مركب 1,5- ثنائي أمينو أنثراكوينون بطريقة الصهر

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#### الملخص

يهدف هذا البحث تحضير مركبات أزو- قواعد شف غنية بالطاقة مشتقة من مركب 1,5- ثنائي أمينو أنثراكوينون وتطوير أطيف الامتصاص لها لكي تكون جزءا مضافا ومكملا لدراساتنا السابقة في مجال تطوير برنامج الخزن الكيماوي للطاقة. لقد تم تحضير خمس مشتقات أزو- قواعد شف من مركب 1,5- ثنائي أنثراكوينون (An1-An5) بالصهر المعتدل لميلليول واحد من المركب المذكور مع ملليمولين من مشتق أزو المحضر من 2- هيدروكسي بنزالديهايد. تم بتطبيق هذه الطريقة الحصول على منتج متوسط الى جيد. أدى استعمال طريقة الصهرالى تحطيم المادة الاولية (الازو)، فيما تم استعادة المواد الاولية بعد ساعات طويلة من التصعيد في مذيب ثنائي مثيل السلفوكسيد.

الكلمات الدالة: 1,5 ثنائي امينو أنثراكوينون، نظم الخزن، تشيع الميكرويف، صهر، اقطاب فائقة السعة.

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## 1. Introduction:

Any proposal to address the energy issue must include competitive, cheap, technically simple and realistic solution. otherwise the problem of impacting the finite fossil energy sources will last, and mankind will continue to suffer from price fluctuations that burden the budget of countries and individuals alike.

Azo-Schiff bases are interesting sort of organic compounds which received the attention of workers in the various fields of applied chemistry for having both effective groups in their backbones: (CH=N) and (N=N) [1-6]. One day after being firstly synthesized at (1977) by R. Botros [1], diligent and probing efforts in diverse approaches concerning promotion of this type of chemicals have been spent. Their multi sites of coordination were precious opportunity for those who carry out researches. Relating to coordination chemistry [7-15]. For researchers interesting in the field of biological activities, it was a chance to test the double effect of their both groups [16-20]. Moreover, the mutual effect of both groups gave workers dealing with spectroscopic studies a wide maneuverability for interpretation [21-23]. And for us who conducting projects concerning photo organic sensitization and contriving new storage systems design, it was almost rarely trodden pathway [24].

1, 5-diaminoanthraquinone (DAAQ) a derivative of anthraquinone dye is chosen to be an ideal molecule to create azomethine moiety holding azo group in its skeleton. The behavior and capacity of (1, 5-diaminoanthraquinone) oligomer as an electrochemical capacitor material was studied [25,26]. In addition the absorption and fluorescence spectral properties of (DAAQ) in some organic solvents with different polarity were investigated [27]. Apart from work registered about the fabrication of poly (1, 5-diaminoanthraquinone) (PDAA) and its application as capacitors and supercapacitors [28-30], very little investigation has been reported relating the organic synthesis taking this dye as starting material [31].

In the recent study five new azo-Schiff bases were synthesized by fusing (2 mmol) of early prepared azo-benzaldehyde compounds with (1 mmol) of 1, 5-diaminoanthraquinone to examine their potent as storage system in the upcoming exertion.

## 2. Experimental:

**2-1 Physical measurements:**Uncorrected melting points of the synthesized compounds were recorded using Stuart-SMP11 melting point apparatus. (UV-VIS) spectrophotometer-PG+92 was used to record the electronic spectra of the synthesized compounds as solutions in DMSO. FTIR spectra ( $\text{cm}^{-1}$ ) were obtained on NICOLT- 100 FTIR Spectrophotometer using KBr discs at the range of (4000-200)  $\text{cm}^{-1}$ .  $^1\text{H}$ NMR spectra (DMSO- $d_6$ ) were performed on NMRReady 60 pro spectrometer taking TMS as internal standard. Elemental analysis was done using Euro EA Elemental Analyzer. The purity of the products and the of the reactions were tracked by thin layer chromatography (TLC) using aluminum sheets of (2 x 4) cm coated with silica gel in benzene-acetone mixed solvent.

**2-2 Chemicals:**All the used chemical and solvents were of reagent grade quality and were purchased from Sigma-Aldrich, BDH, GCC and Schlwa companies.

**2-3 Synthesis procedures:** (1 mmole) of 1, 5-diaminoanthraquinone was mixed with (2 mmole) of the early prepared compounds (Ia-e) [18]. in a beaker using a glass rod until it became moistened. Afterwards, some drops of ethanol were added and the mixture was gently heated for (15-20) minutes. Heating was stopped when the color and statue of the mixture changed. The cooled products were washed with petroleum ether (60-80) °C several times, then filtered and recrystallized from ethanol; compound An<sub>5</sub> was washed with hot ethanol. The reaction was monitored by TLC (silica gel plates with iodine indicator).

**An<sub>1</sub>:** C<sub>40</sub>H<sub>26</sub>N<sub>6</sub>O<sub>4</sub>; 1,5-bis((2-hydroxy-4-(phenyldiazenyl)benzylidene) amino)anthracene-9,10-dione; Brown powder, m.p.: 120-123 °C, Yield: 80%; C, H, N:found/(calc.) C: 73.14(73.39), H:3.65(3.97), N: 12.59 (12.84); ( FTIR (KBr,  $\text{cm}^{-1}$ ): 3400 (O-H), 3000 (C-H Ar.) 1655 (C=O), 1621 (C=N), 1580 (C=C Ar.), 1458 (N=N);  $^1\text{H}$ NMR (DMSO- $d_6$ ):  $\delta$ =7.0-8.50 (m, 22 H, ArH), 10.30 ( s, 2H, HC=N), 11.47 (s, 2H, OH) ppm; UV-Vis:  $\lambda_{\text{max}}$ = 225,340, 498 nm.

**An<sub>2</sub>:** C<sub>42</sub>H<sub>30</sub>N<sub>6</sub>O<sub>4</sub>; 1,5-bis((2-hydroxy-4-(p-tolyldiazenyl) benzylidene) amino) anthracene-9,10-dione; Dark Brown powder, m.p.:288-290 °C, Yield: 82%; C, H, N:found/(calc.) C: 73.45(73.90), H:4.65(4.39), N: 12.42 (12.31); FTIR (KBr,  $\text{cm}^{-1}$ ): 3410 (O-H), 3030 (C-H Ar.), 2850 (C-H alp.), 1657 (C=O), 1616 (C=N), 1585 (C=C Ar.), 1458 (N=N);  $^1\text{H}$ NMR (DMSO-

d<sup>6</sup>):  $\delta=3.47$  (s, 6H, 2CH<sub>3</sub>), 7.60-8.05 (m, 26 H, ArH), 10.38 (s, 2H, HC=N), 11.50 (s, 2H, OH) ppm; UV-Vis:  $\lambda_{\max}=225,340,490$  nm.

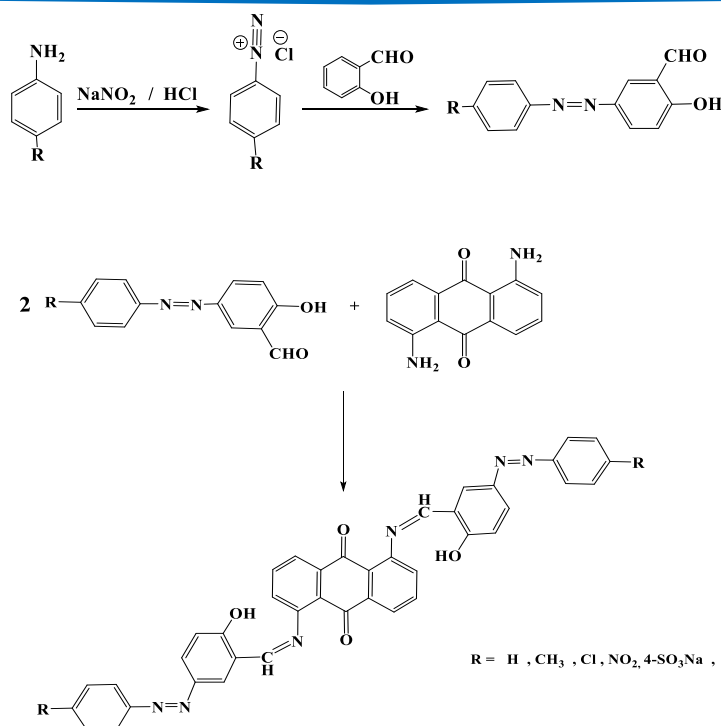
**An<sub>3</sub>:** C<sub>40</sub>H<sub>24</sub>N<sub>6</sub>O<sub>4</sub>Cl<sub>2</sub>; 1,5-bis ((4-(4-chlorophenyl) diazenyl)-2-hydroxy benzylidene)amino)anthracene-9,10-dione; Reddish Brown powder, m.p.: 280-282 C, Yield: 78%; C, H, N:found/(calc.) C: 67.45(67.03), H:4.29(3.35), N: 11.49 (11.73); FTIR (KBr, cm<sup>-1</sup>): 3980 (O-H), 3020 (C-H Ar.) 1656 (C=O), 1620 (C=N), 1584 (C=C Ar.), 1448 (N=N), Cl (C-Cl); UV-Vis:  $\lambda_{\max}=227,343,495$  nm.

**An<sub>4</sub>:** C<sub>40</sub>H<sub>24</sub>N<sub>8</sub>O<sub>8</sub>; 1,5-bis((2-hydroxy-4-((4-nitrophenyl) diazenyl) benzylidene) amino) anthracene-9,10-dione; Dark Brown powder, m.p.: 235-240 °C, Yield: 77%; C, H, N:found/(calc.) C: 64.01(64.51), H:3.45(3.22), N: 15.39 (15.05); FTIR (KBr, cm<sup>-1</sup>): 3400 (O-H), 3010 (C-H Ar.) 1655 (C=O), 1622 (C=N), 1590 (C=C Ar.), 1480 (N=N), 1341, 1514 (NO<sub>2</sub> group); UV-Vis:  $\lambda_{\max}=244,350,500$  nm.

**Ans:** C<sub>40</sub>H<sub>24</sub>N<sub>6</sub>O<sub>10</sub>Na<sub>2</sub>S<sub>2</sub>; sodium 4, 4'-((((9,10-dioxo-9,10-dihydroanthracene-1,5-diy)bis(azanylidene))bis(methylydene))bis(3-hydroxy-4,1-phenylene))bis(diazine-2,1-diy)l) dibenzenesulfonate; Reddish Brown, m.p.: > 300 °C, Yield: 75%; FTIR (KBr, cm<sup>-1</sup>): 3422 (O-H), 3025 (C-H Ar.) 1659 (C=O), 1612 (C=N), 1585 (C=C Ar.), 1493 (N=N), 1170, 1355 (SO<sub>2</sub> group); UV-Vis:  $\lambda_{\max}=225,320,495$  nm.

### 3. Results and Discussion:

Scheme (1) exhibits the synthesis pathway of the azo-Schiff compounds (An<sub>1</sub>-An<sub>5</sub>) in two successive steps. The well-known starting materials (Ia-e) are reproduced in the first step following the procedures in the literature [18]. The second step shows the synthesis of the title compounds. It is noticed that the desired products can only be acquired by gentle fusion method. Since some azo compounds are unstable at elevated temperature, [24] the starting material (2-hydroxy azo-benzaldehydes) decomposes due to irradiation in microwave reactor. On the other hand, under five days of reflux in DMSO, the starting materials would be recovered. This result is attributed to the potent dipole-dipole effect of the used aprotic solvent with the lone pairs of amino groups, which provides an extra reason that shows that they are inefficacious [21]. The purity of the macromolecule prepared was tested by CHN analysis and thin layer chromatography.



**Scheme 1:** Preparation of azo-schiff base compounds.

### 3-1 Electronic spectra

The UV-visible spectra of ( $1 \times 10^{-4}$  M) solutions of the synthesized compounds (An<sub>1</sub>-An<sub>5</sub>) were recorded in dimethylsulfoxide (DMSO). The general pattern of the tested compounds showed the following main peaks: the first one around (225-227) nm due to the medium ( $\pi$ - $\pi^*$ ) transition with high absorptivity value. This peak, in its shape and location is similar to those sourced from first transition of Schiff bases around (236 nm) due to conjugation of lone pair of electrons on nitrogen atom with the adjacent aromatic ( $\pi$ ) system. The second peak at (320-350) nm due to the lower ( $\pi$ - $\pi^*$ ) of the azomethine group, and the third peak at (490-500) nm due to lower absorption value of ( $n$ - $\pi^*$ ) transitions of the azo group may be overlapped with the same transition of carbonyl groups [31,32]. Noticeable improvement in the broadness and strength of the third peak of the products spectra, comparing with the starting material (DAAQ) was observed in consistent with our prim interest. Hence, the electronic spectra of some amines have been modified by incorporating azomethine groups in their skeleton, which, consequently, has produced some photostable sensitizers [21,33]. Therefore, the synthesized compounds are

expected to be efficient, and energy-rich; the properties that make them suitable to be used in photochemical storage systems.

### 3-2 FTIR spectra:

The disappearance of the characteristic bands ascribed to the stretching vibrations of (N-H) bond of the diamino groups of the 1, 5-diaminoanthraquinone at (3477  $\text{cm}^{-1}$ ) and (3381-3204)  $\text{cm}^{-1}$  in addition to the shift in the location of intra hydrogen bonded carbonyl group from (1609  $\text{cm}^{-1}$ ) of the starting material to (1655-1659)  $\text{cm}^{-1}$  are strong evidences for the development of the product. The appearance of new sharp bands at (1612-1620)  $\text{cm}^{-1}$  referring to (CH=N group) indicates the formation of Schiff bases [7-18]. Moderate bands at (1580-1595)  $\text{cm}^{-1}$  referring to the stretching absorption of aromatic (C=C) bond and weak bands (1017-1060)  $\text{cm}^{-1}$  due to (N=N) group are another evidence for the formation of the desired product. The diversity in the location of the newly formed main bands (CH=N, N=N) is ascribed to the impacts of the substitution groups at the terminal aromatic rings Fig. 1,2 show the infrared spectrum of the compounds An1 and An4 respectively.

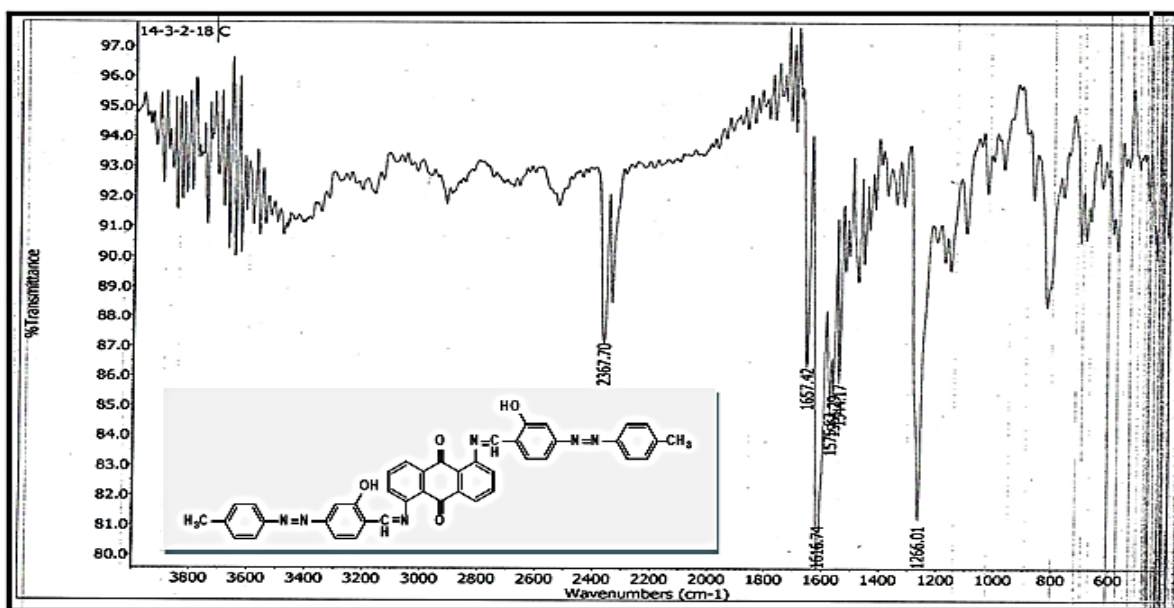


Fig. 1: Infrared spectrum for An2.

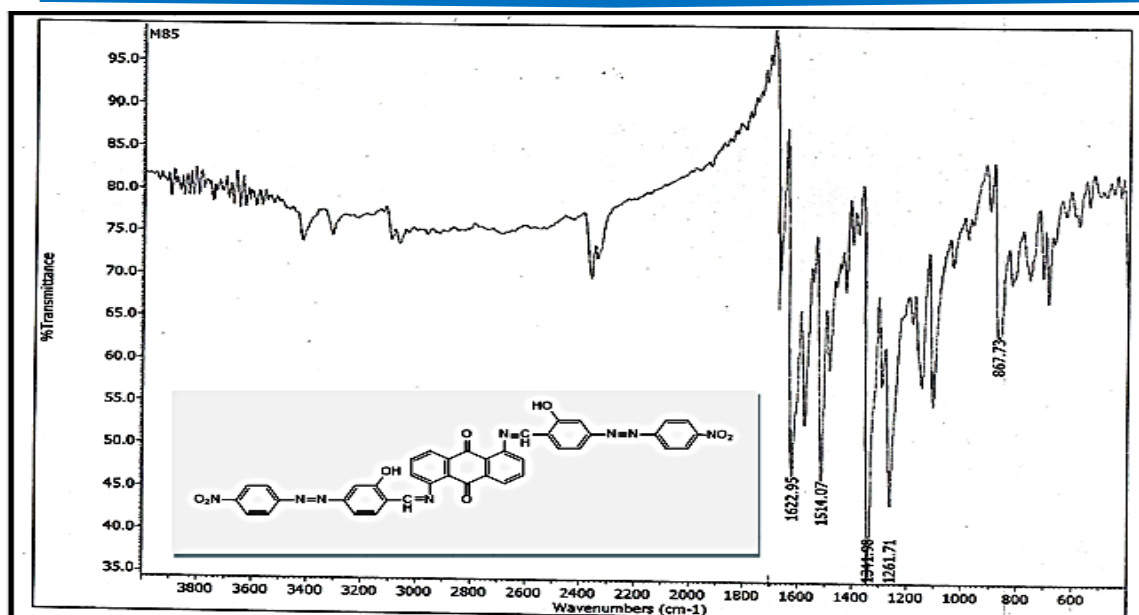


Fig. 2: Infrared spectrum for An4.

### 3-3 <sup>1</sup>HNMR spectra:

The general feature of the <sup>1</sup>HNMR spectrum recorded for the prepared compounds reveals the appearance of a multiple signal at (7.0-8.5) ppm for the aromatic protons of the whole compound. Single signals at (10.5) ppm for hydroxyl protons in addition to single signal at (11.4) ppm for azomethane protons were also recorded. Extra signals exhibited at different position of the spectra are attributed to the various substituents at the aromatic rings Fig. 3 exhibits the proton NMR spectrum of the compound An1 .

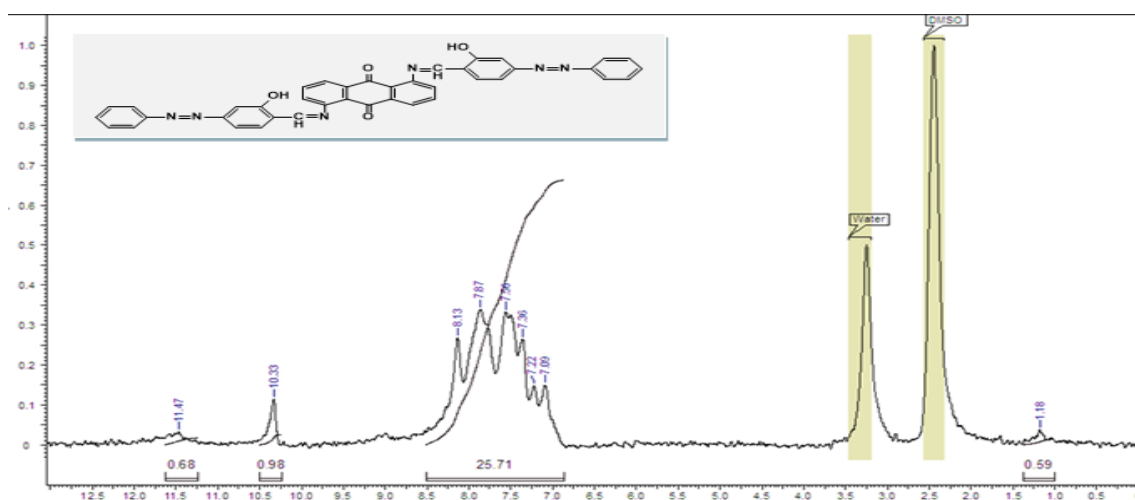


Fig. 3: Proton NMR spectrum for An1.



#### 4. Conclusions:

Synthesis of azomethine of 1, 5-diaminoanthraquinone dye has led to extend and improve their electronic characterizations, which may serve to be a suitable candidate in a future work regarding photochemical reactions. Moreover, microwave irradiation of azo compounds has shown to cleave the (-N=N-) bond, resulting in their dissociation.

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