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Design, Synthesis and Structural Characterization of Novel Thioanthraquinone Analogues from 1,5-Dichloroanthraquinone

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Abstract: Anthraquinone and its derivatives are considered intermediate agents with superior properties due to their activities in chemical and biological reaction. A new, economical, practical and one-step synthesis method was developed by our research team for the synthesis of amino and thioanthraquinones in previous studies (1). With this synthesis method, thioanthraquinone analogs **2(a-d)** were obtained from 1,5-Dichloroanthraquinone **(1)** and bioactive thiols. The synthesized organic molecules were purified by column chromatography and their structures were identified with spectroscopic methods. Fluorescence analyzes of synthesized thioanthraquinone analogues were performed. It was determined that all thioanthraquinone analogues synthesized and characterized in the study showed fluorescence activity. These new analogues with fluorescence are expected to find application in drug delivery systems and sensor studies.

Keywords: Anthraquinone, fluorescence, organic synthesis, spectroscopic analysis, thiols.

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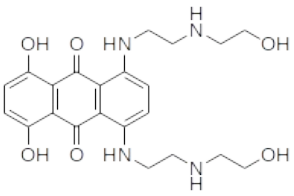
1. INTRODUCTION

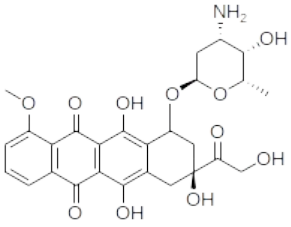
Cancer has still been at the top of the list of diseases that threaten human health and may result in death (2). Anthraquinones are one of the most active members of organic compounds. Anthraquinone and its analogs, which are biologically active, have antimicrobial, antifungal, antioxidant, anti-cancer, antitumor, anti-HIV, anti-Alzehiemer and anti-inflammatory properties (3-9). An amino anthraquinone derivative, mitoxantrone, which is one of the important anticancer drugs and accepted as an antineoplastic agent, is known to have effects against tumors in cancer types such as ovarian, breast, prostate, leukemia, and lymphoma (10-12) (Figure 1). Mitoxantrone, a topoisomerase II inhibitor found to be effective in the treatment of various tumors and multiple sclerosis disease, has been found to have potential neurotoxic effects (13).

In a study, Pan et al. (14) isolated anthraquinone derivatives as a secondary metabolite from the saprophytic sea fungus Alternaria tenuissima, which causes skin infections in some people (14-20). Wang et al. showed that water-soluble anthraquinone derivatives with antitumor properties killed tumor cells in the apoptotic pathway against gastric cancer cells (21-22). Anthraquinones have a redox system due to their acid-base properties (23) and they are reduced to hydroquinones under protic conditions (24-25). Hydroquinone is oxidized in the redox structure and becomes anthraquinone again (24-25). The capability of energy storage of quinones and especially the anthraquinones, allows them to be preferred as organic material in these redox flow systems (26-28). Although various derivatives of anthraquinones are found in the literature, studies on thio-anthraquinone analogs are limited. A new, practical and economical synthesis method for the synthesis of amino and thio-anthraquinone molecules has been developed in previous studies of our team (1). Various amino and thio-anthraquinone analogues were synthesized with this new synthesis method (29-31). It was determined that these amino and thioanthraquinone derivatives synthesized in previous studies show anticancer and antimicrobial activities (29-31). In a study by Şahin, Y.M. (32) an antimicrobial thioanthraquinone analogue was synthesized and a nanobiocomposite was obtained

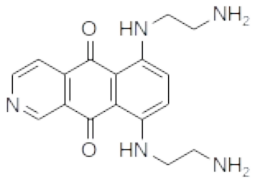
from this thioanthraquinone analogue for use in tissue engineering applications. The aim of this study is to synthesize bioactive 1-substituted-5-chloro derivatives that can be used in health, medicine and materials fields and to characterize their structures.



Mitoxantrone



Doxorubicin



Pixantron

Figure 1. Anthraquinone analogues as anti-cancer drugs (4).

2. EXPERIMENTAL SECTION

2.1. Reagents and Instruments

All reagents used were obtained from Sigma-Aldrich. In the infrared (FT-IR) spectra, the Shimadzu FTIR-8101 spectrometer was used. Varian^{UNITY} INOVA 500 MHz device operating in NMR analysis was used. Mass spectrum analyzes were accordingly performed on a Thermo Advantage MAX LC/MS/MS instrument using APCI or ESI methods. Florescence spectra were recorded using Spectro-SpectroBlue fluorescence spectrophotometer. Buchi B-540 melting point device was used for melting point analysis. Silica gel (Fluka Silica gel 60, particle size 63-200 µm) was used for purification in column chromatography of the synthesized analogues. Silica 60F254 (Merck, Darmstadt) was used for TLC layers in the study.

2.2. General synthesis method (2a-d):

All novel anthraquinone analogues **2(a-d)** were obtained from the reaction of 1,5-Dichloroanthraquinone **(1)** and bioactive thiols according to the general synthesis method (1) for anthraquinone analogues. One equivalents molar of 1,5-Dichloro-9,10-anthrecenedione **(1)** and thiols were stirred in 110-120 ˚C a mixture of ethylene glycol (30 ml) and aqueous solution of KOH (1.2 g KOH and 8 ml water) for 48h with reflux system. Chloroform was added to the reaction mixture to separate the organic layer. Then, the organic layer was washed with water (4x30 ml) and dried over Na_2SO_4. After filtering, the solvent was evaporated and the residues (**2a-d**) was purified by column choromatography on silicagel (Scheme 1).

*2.2.1. 1-Chloro-5-(Dodecylthio)anthracene-9,10-dione **(2a)***

Yellow solid thioanthraquinone compound **2a** was obtained from the reaction of 1,5-Dichloroanthraquinone **(1)** and 1-Dodecanethiol according to the general synthesis method.

Dark yellow semi-solid. Yield: 0.37 g (23%). R_f (1 Petrolium ether / 1 Chloroform): 0.46. IR (KBr, cm^{-1}): υ = 2980, 2962, 2952 (C-H_{arom}), 2938, 2915, 2846 (C-$H_{alifatic}$), 1671 (C=O), 1573 (C=C). ^{1}H NMR (499.74 MHz, $CDCl_3$): δ = 7.26-8.27 (m, 6H, CH_{arom}) , 2.97-3 (t, 2H, *J=2.98*, -S-*CH_2*), 1.26-2.23 (m, 20H, CH_2), 0.86-0.88 (t, 3H, *J= 0.87*, CH_3). ^{13}C NMR (125.66 MHz, $CDCl_3$): δ = 183.1 (C=O), 159.4, 145.2, 136.9, 136, 135.3, 133, 129.3, 123.3, 121, 120.3 (C_{arom} and CH_{arom}), 14.1 (CH_3). ESI(+): *m/z*= 461.1 $[M+NH_4^+]^+$, 274,85 $[M-S(CH_2)_{11}CH_3]^+$. (M= 443.04 g/mol). $C_{26}H_{31}ClO_2S$, (M, 443,04 g/mol).

*2.2.2. Butyl-3-(1-Chloro-9,10-dihydro-9,10-dioxoanthracen-5-ylthio)propionate **(2b)***

Orange semi-solid thioanthraquinone compound **2b** was obtained from the reaction of 1,5-Dichloroanthraquinone **(1)** and Butyl-3-Mercaptopropionate according to the general synthesis method.

Orange semi-solid. Yield: 0.43 g (32%). R_f (Ethyl acetate): 0.45. IR (KBr, cm^{-1}): υ = 3464 (C=O_{ester}), 2954, 2915, 2870, 2850 (C-H_{arom}), 1658 (C=O), 1651 (C=C). ^{1}H NMR (499.74 MHz, $CDCl_3$): δ = 7.26-8.03 (m, 6H, CH_{arom}) , 4-4.3 (m, 2H, COO-*CH_2*), 2.97-2.99 (t, 2H, *J=2.98*, S-CH_2), 1.24-2 (m, 6H, CH_2), 0.92-0.94 (t, 3H, *J= 0.93*, CH_3). ^{13}C NMR (125.66 MHz, $CDCl_3$): 183, 183.1 (C=O), 159.4, 145.2, 136.9, 136, 135.3, 133, 129.2, 127.9, 123.3, 121.5, 120.9, 120.2 (C_{arom} and CH_{arom}), 72 (COO*CH_2*), 32,31.4, 27.5, 22.3 (CH_2), 13.9 (CH_3). MS [-ESI]: *m/z* = 309.55 $[M-C_4H_9-Cl]^+$. $C_{21}H_{19}ClO_4S$, (M, 402.89 g/mol).

*2.2.3. 1-Chloro-5-(pentylthio)anthracene-9,10-dione **(2c)***

Yellow solid thioanthraquinone compound **2c** was obtained from the reaction of 1,5-Dichloroanthraquinone **1** and Pentanethiol according to the general synthesis method.

Yellow solid. Mp: 187-188 °C. Yield: 0.22 g (17%). R_f (Ethyl acetate): 0.43. IR (KBr, cm^{-1}): υ = 3065 (C-H_{arom}), 2954, 2915, 2870, 2850 ($CH_{aliphatic}$), 1726 (C=O), 1651 (C=C). ^{1}H NMR (499.74 MHz, $CDCl_3$): δ = 7.26-8.05 (m, 6H, CH_{arom}) , 4.03-4.32 (m, 2H, S-*CH_2*), 1.37-3 (m, 6H, CH_2), 0.92-0.95 (t, 3H, *J= 0.93*, CH_3). ^{13}C NMR (125.66 MHz, $CDCl_3$): δ = 183.2 (C=O), 120.3, 121, 121.5, 123.3, 129.3, 133.1, 135.3, 136, 136.9, 145.2, 159.4 (C_{arom} and CH_{arom}), 60.7 (S-***C***H_2), 22.3, 27.5, 31.4, 32 (CH_2), 13.9 (CH_3). MS [-ESI]: *m/z* = 368.90 $[M+Na^+]^+$. $C_{19}H_{17}ClO_2S$, (M, 344,86 g/mol).

*2.2.4. 1-(4-Fluorophenylthio)-5-chloroanthracene-9,10-dione **(2d)***

Yellow solid thioanthraquinone compound **2d** was obtained from the reaction of 1,5-Dichloroanthraquinone **1** and 4-Fluorothiophenol according to the general synthesis method.

Yellow solid. Mp: 191-192 °C. Yield: 0.28 g (21%). R_f (Ethyl acetate): 0.44. IR (KBr, cm^{-1}): υ = 3465, 3391, 3319, 3065, 2938, 2878 (C-H_{arom}), 1671 (C=O), 1578 (C=C). ^{1}H NMR (499.74 MHz, $CDCl_3$): δ = 7.27-8.25 (m, 10H, CH_{arom}). ^{13}C NMR (125.66 MHz, $CDCl_3$): δ = 183.1 (C=O), 120.8, 121, 122.5, 126.7, 127.3, 132.5, 133.5, 134.3, 134.8, 135.1, 135.4, 159.8 (C_{arom} and CH_{arom}). MS [-ESI]: *m/z* = 393,05 $[M+Na^+]^+$. $C_{20}H_{10}ClFO_2S$, (M, 368,81g/mol).

2.3. Fluorescence Spectroscopy

All novel thioanthraquinone analogues synthesized in the study showed fluorescence properties. Delocalization of π-bond in the aromatic thio-anthraquinone skeleton is effective in determining the fluorescence characteristic of molecules. Excitation and emission wavelengths in the fluorescence spectrum of the **2a** molecule were observed as 530 nm (λexc.) and 565 nm (λem.). Excitation and emission wavelengths in the fluorescence spectrum of the **2b** were observed as 518 nm (λexc.) and 567 nm (λem.). Excitation wavelengths in the fluorescence spectrum of the **2c** and **2d** were observed as 580 nm (λexc.) ve 505 nm (λem.), respectively. The fluorescence graphic of all synthesized novel thioanthraquinone analogues is demonstrated in Figure 2.

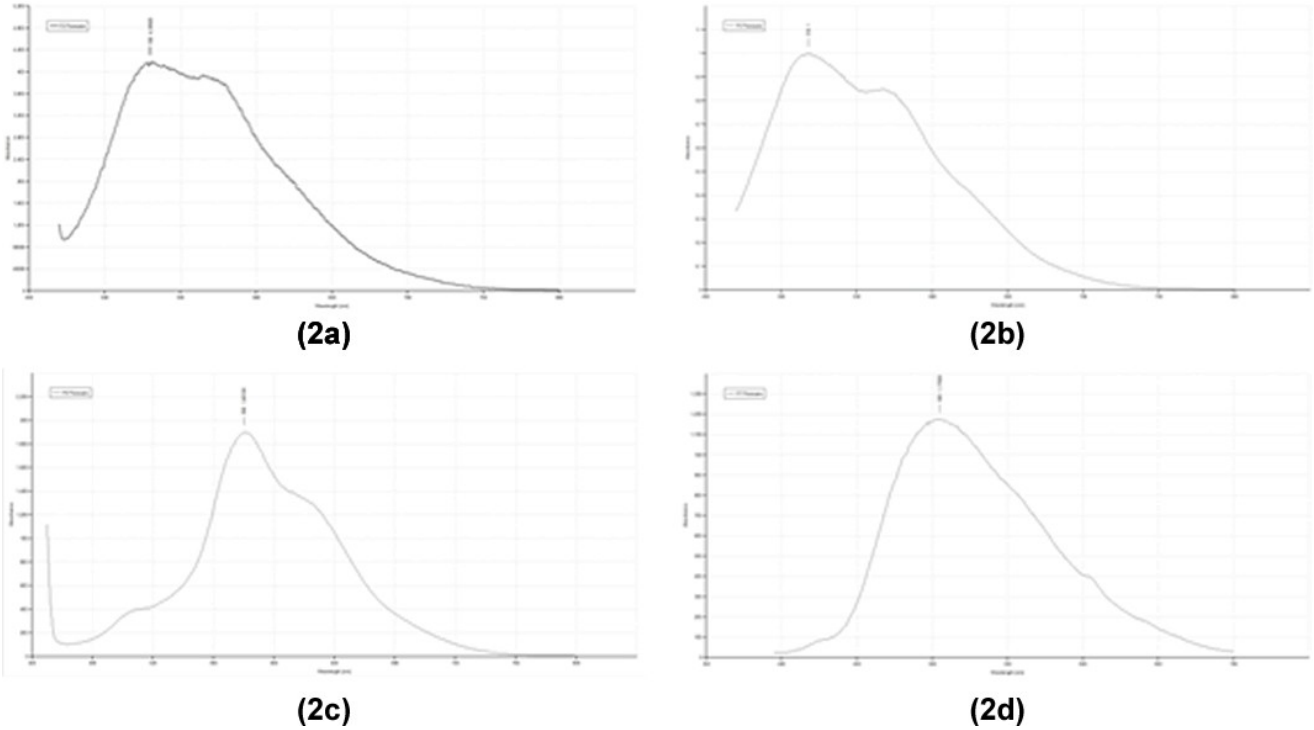
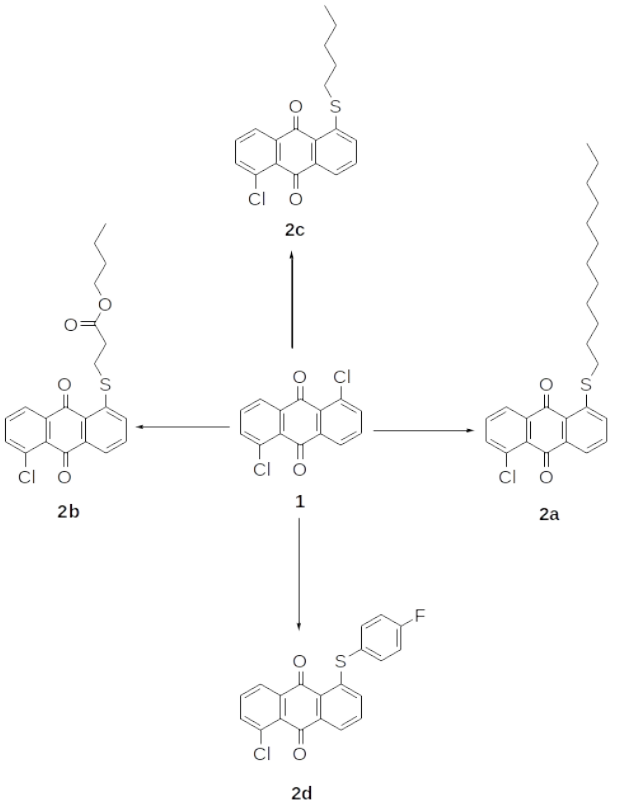


Figure 2. The fluorescence graphs of all synthesized novel thioanthraquinone analogues **(2a-d)**.

3. RESULTS AND DISCUSSION

In the first step of the study, 1-chloro-5-(dodecylthio)anthracene-9,10-dione **(2a)** was synthesized from the reaction of the starting material 1,5-Dichloro-9,10-anthracenedion **(1)** and Dodecanethiol according to the general procedure. In the second step, Butyl-3-(1-Chloro-9,10-dihydro-9,10-)dioxoanthracen-5-ylthio)propionate **(2b)** was obtained from the reaction of 1,5-Dichloro-9,10-anthracenedione **(1)** and butyl-3-mercaptopropionate according to the general procedure. In the third step, 1-chloro-5-(pentylthio)anthracene-9,10-dione **(2c)** was synthesized from the reaction of the starting material 1,5-dichloroanthraquinone **(1)** and 1-pentantiol according to the general procedure. In the last step of the study, 1-(4-fluorophenylthio)-5-chloroanthracene-9,10-dione **(2d)** was obtained from the reaction of the starting material 1,5-dichloroanthraquinone **(1)** and 4-fluorothiophenol according to the general procedure. All synthesized thioanthraquinone analogues are presented in Scheme 1.



Scheme 1: Distribution of novel thioanthraquinone analogues.

In the FTIR spectra of synthesized thioanthraquinone derivatives **2(a-d),** characteristic absorptions belonging to the C=O group were seen at 1671, 1726 and 1671 cm^{-1}. In the ^{1}H-NMR spectrum, the CH_3 group peaks of the **(2a)** molecule were confirmed to be triplets between 0.86-0.88. Observation of the sulfur-bound CH_2 group in the molecule to be a triplet between 2.97-3 and the other aliphatic CH_2 groups to be multiplets between 1.26-2.23 confirms the structure of the thio-anthraquinone molecule **(2a).** The carbonyl group peaks in the ^{13}C-NMR spectrum of the synthesized analogues were confirmed at 183, 183.1, 183.2, respectively. While CH_2 bound to the ester group was observed at 72 by shifting to down domain in the ^{13}C-NMR spectrum of the **(2b)** molecule, other CH_2 groups in the chain were observed at 32, 31.4, 27.5 and 22.3, respectively.

4. CONCLUSION

Anthraquinone and its derivatives play an important role in chemical and biological reactions thanks to the redox system in their structure. At the same time, the electron exchange of anthraquinone and its derivatives in this redox system allows them to be active in energy storage systems. Although there are various studies on the synthesis and biological activities of amino anthraquinone analogs in the literature, studies on the synthesis, biological activity properties and material properties of new thioanthraquinone molecules are quite limited. The new molecules obtained via the new synthesis method developed and patented by our team for the synthesis of amino and thio-anthraquinones can be considered as significant agents for reactions. The fluorescence aspects of the synthesized molecules render these molecules to be interesting in sensor studies. It is predicted for these novel thioanthraquinone molecules synthesized in this study that they may provide a new perspective to studies in the fields of chemistry, biology, medicine, pharmacy, material science and renewable energy.

5. CONFLICT OF INTEREST

The authors declare that there are no conflict of interests.

6. ACKNOWLEDGMENTS

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