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Antioxidant Activity of some Dibenzylidine Diamine-18-Crown-6 Materials

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ABSTRACT

In the present study,Dibenzylidine Diamine-18-Crown-6 materials have been synthesised by the condensation of Diamine-18-Crown-6 with subsituted benzaldhyde and characterized by Elemental Analysis, Infra red and Proton Magnetic Resonance Spectroscopy.All the synthesized materials were screend for their antioxidant behaviour using standared DPPH free radical as hydrogen acceptor.

Keywords: Antioxidant,Free radical scavenger, Benzylidine, Crown ethers.

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INTRODUCTION

Reactive free radicals such as O_2^-, $OH^\cdot$ and $CH_3^\cdot$, induce oxidative damage to healthy cells of the body due to their ability to initiate chain reactions which lead to the propogation of chemical modification of polyunsaturated membrane lipids, proteins and nucleic acids[1-5]. Benzylidin containing compounds have stimulated interesting research in synthetic Chemistry, Biology and Pharmacology. They exhibt a variety of pharmacological activities including antiinflammatory[6], anticarcinogenic[7], antibacterial[8,9] and antifungal [10,11] activities most of which are attributed to their antioxidant properties

Due to our knowledge, very few medicinal studies were carried out on macro molecules containing benzylidine functional groups[12]. In this paper, we focused our study on preparation, characterization and antioxidant properties of some macro dibenzylidine. Presently, synthetic antioxidants are widely used because they are cheaper than natural antioxidants.

MATERIALS AND METHODS

All chemicals and solvents used were of analytical grade. Infra red spectra were recorded using KBr disc on a FT-IR spectrometer, shimadzu 8400s in the range of 4000-400 cm^{-1} . ^{1}H-NMR spectra were recorded in $CDCl_3$ at room temperature. The elemental analysis was performed at elemental analyzer Perkinelmer CHNS-O analyzer model 2400 series II. Column chromotography was carried out with silica Gel powder (30-70 mesh size) by using appropriate solvents.Geometrical optimization of dibenzyzlidine diamine-18-crown-6 materials was drawn by using PM3 method.

EXPERIMENTAL

Preparation of dinitro dibenzo-18-crown-6 (DNDB 18C6)

This compound was prepared[13] by mixing of dibenzo-18-crown-6 (5 gm, 0.13 mole), 60 ml chloroform and 75 ml glacial acetic acid followed by a solution of (3.5 ml, 0.57 mole) concentrated nitric acid in 10 ml acetic acid. The reaction was stirred without heating for an hour, during which it successively turn green, blue and yellow. After mixing on heating mantle for three hours, the product was filtered and recrystallized from dimethyl formamide (DMF), then dinitro dibenzene-18-crown-6 was finally obtained.

Preparation of diamino dibenzene-18-crown-6 (DADB 18C6)

3 gm of dinitro dibenzo-18-crown-6 was added to 75 ml distilled water.To this slurry product, 15 gm of sodium sulfide nine hydrate was added and stirred at 60^{o}C for 24 hrs. The product was filtered and washed with 50 ml 1N hydrochloric acid then treated with 50 ml of 1N sodium hydroxide. The product was washed with distilled water until the product was free from NaOH and NaCl. The crude product of diamino dibenzene-18-crown-6 was finally recrystalized from ethanol[14].

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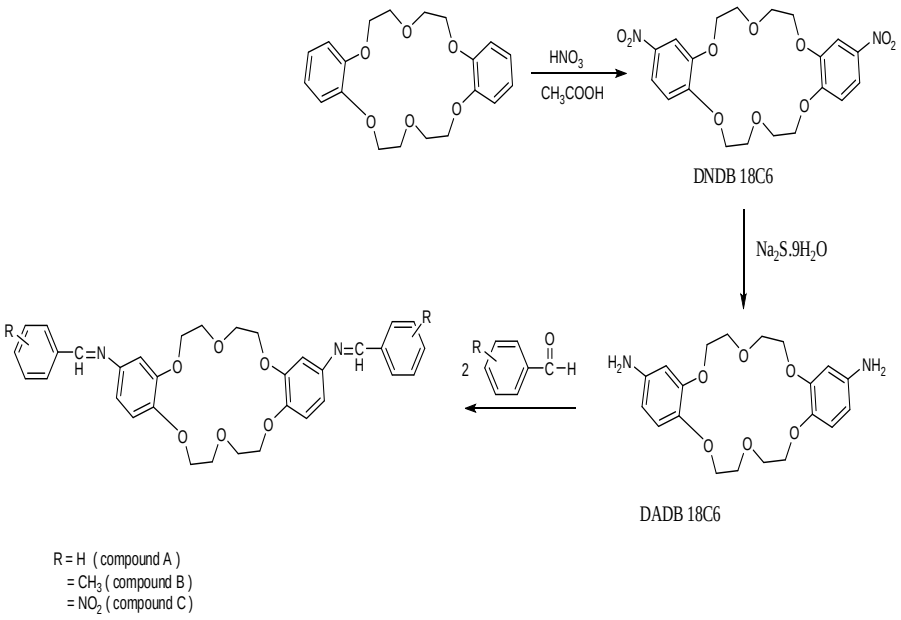
Synthesis of dibenzylidene diamine-18-crown-6 materials

A reaction vessel was charged with (0.005 mole) of diamino dibenzene-18-crown-6 in (5 ml) of dry pyridine , and the mixture was vigrously stirred. Solution of (0.01 mole) of Benzyldhde or para subsituted benzyldhyde in (5 ml) of dry pyridine was added. The vessel was tightly closed and the reaction was kept at room temperature with stirring for 24 houres. The solution was neutralized with (10%) hydrochloric acid,then the product was filtered and washed several times with distilled water then dried. The final product of dibenzylidine diamine-crown-6 was recrystallized several
times from ethanol.

Scheme (1) shows the synthesis routes of the dibenzylidene diamine-18-crown-6 Proudcts.



Scheme 1: Synthesis routes of the dibenzylidine diamine-18-crown-6 products(A),(B) and (C).

RESULTS AND DISCUSSION

Characterization of dibenzylidine diamine-18-crown-6

The structure of the dibenzylidine diamine-18-crown-6 compounds were characterized by elemental analysis, infra red and nuclear magnetic resonance spectroscopy. The elemental analysis data are in good agreement with calculated values as shown in Table(1).The appearance of absorption band at 1623-1630 cm^{-1} which is characterstic of C=N stretching vibration, the disappearance of both bands at 3400 and at 3500 cm^{-1} which is characterstic of symetric and asymetric stretching vibrations for NH_2 group together with disappearanc of band at1695 cm^{-1} which characterstic of C=O stretching vibration all indicate that the reaction of amine and benzaldhyde to obtain the dibenzylidine diamine-18-crown-6 is successful.The most important vibration bands

observed in infrared spectra for the prepared compounds are shown in Table(2). The ^{1}H-Nnclear magnetic resonance spectra of dibenzylidine diamine-18-crown-6 compounds showed broadly similar spectral characteristic . The main feature of these spectra is the apperance of low field resonance for the proton of azomethane group which gives broad signal at 7.8 ppm , the protons of phenyl ring fused with crown ether give multiplet signals in the range 7.7 -7.8 ppm, while the protons of the Benzene ring give multiplet in the range 6.7-6.9 ppm. The 8 protons of CH_2O-ph give triplet signals at 4.2 ppm, while the 8 protons of CH_2O give triplet signals at 3.7 ppm.

Table 1: Elemental analysis of dibenzylidine diamine-18-crown-6

	Found			Calculated		
Compound	C	H	N	C	H	N
A	71.85	6.12	4.89	72.08	6.01	4.95
B	71.98	6.19	5.02	72.41	6.21	4.82
C	70.01	5.61	6.88	69.03	5.58	7.10

Table 2 : The location of most of the vibration bands (cm^{-1}) for dibenzylidine diamine-18-crown-6

Compound	C-H str. Aromatic	C-H str. Aliphatic	C=C str. Aromatic	C=N str.	C-O str. Ether
A	3100	2920-2900	1595	1623	1270-1140
B	3096	2921-2902	1599	1630	1270-1140
C	3103	2922-2904	1596	1628	1272-1142

Antioxidant Activity of dibenzylidine diamine-18-crown-6

The synthesized dibenzylidine diamine-18-crown-6 compounds were subjected to their possible antioxidant activity using DPPH free radical as hydrogen acceptor. DPPH absorbs visible light at 517 nm and,as antioxidants donate protons to this radical, the absorption decreses. Figure 1 shows the antioxidant activity of the dibenzylidine compounds A, B, and C after 1 hour of incubation. The percentage of antioxidant activity was calculated using the following equation [15]:-

Antioxidant activity(%) = Ao-As/AoX100

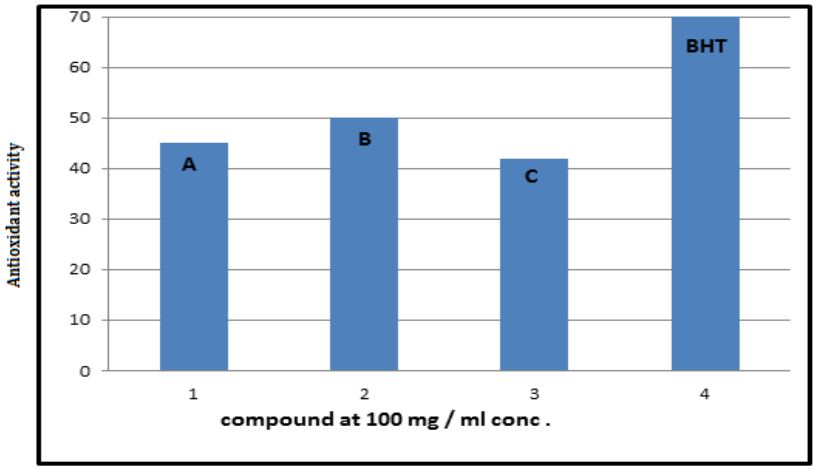


Figure 1: Antioxidant activity of the prepared compounds (A,B,C)

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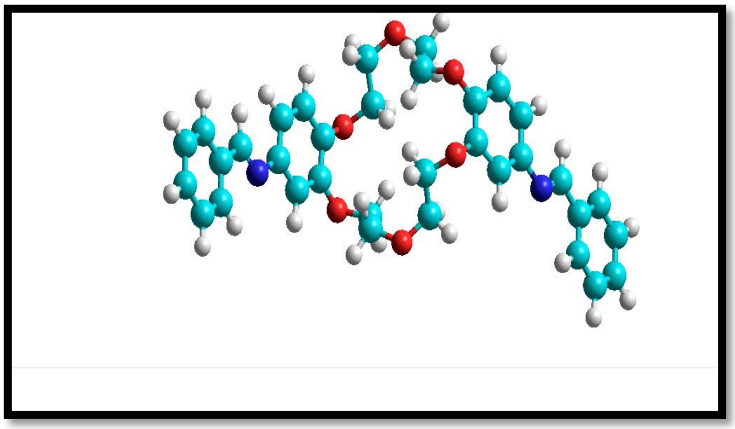

Figure 2: Optimized 3D structure of dibenzylidine diamine-18-crown-6

Where Ao is the absorbance of the control and As is the absorbance of the test sample or standard (BHT). As shown in figure 1, the dibenzylidine diamine-18-crown-6 componds show a pronounce antioxidant activity compared to that of butylated hydroxyl toluene (BHT). Compounds A,B and C have slightly flactuated antioxidant activity due to the nature of R subistuent attached to dibenzylidine moeity.The subsituent with electron donating (NO_2) group shows slightly lower antioxidant behaviour corresponding to subsituent with electron withdrawing (CH_3) group.bThe geometrical optimization of dibenzylidine diamine-18-crown-6, shown in figure 2indicate that the release of hydrogen atom from benzylidene to the radical undergoes quite easily.

CONCLUSION

The present study demonstrated that the synthesized macromolecules containing dibenzylidine diamine-18-crown-6 exhibited feasible antioxidant activity.The strength of antioxidant behaviour of these materials depends on the geometry of the molecule and the type of subsituent group attached to benzylidine moeity.

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