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Synthesis of New Heterocyclic Compounds by Diels - Alder Reaction using Ultrasound Technique

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Abstract: ***A new heterocyclic compounds were synthesized by Diels- Alder reaction using ultrasound technique. A reaction between bezaladehyde and several aromatic compounds formed compounds (9, 10, 11) which identified by infrared spectra (FT-IR), 1HNMR and CHN analysis. The prepared compounds manifested promising biological activity.***

Keywords: benzaldehyde, Diels –Alder, heterocyclic, oxo compounds.

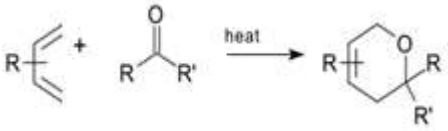
1. Introduction

Diels-Alder (DA) reaction has been around for a very long time, it's the most known cyclo addition reactions. This reaction has great influence on the future study and treatment of diseases [1], as a very great strength method for manufacturing an unusual compounds having possible action on tumors [2].

It is particular impact on complex targeted natural product synthesis arises, in the first instance, from its capacity to deliver extensively functionalized six-membered rings with defined sites of unsaturation and functional group placements. Moreover, it provides the basis for coordinating stereogenic centers in the emerging cyclohexene. These configurational relationships are governed by the apparently universal superficial nature of the Diels–Alder reaction [3, 4].

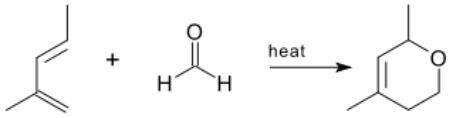
(DA) reactions involve electron-rich dienes and electron-poor dienophiles, and under certain conditions; the reverse reaction is known as the retro-Diels–Alder reaction[5]. The general reaction involves a conjugated diene and a dienophile [6, 7] as shown below:

Diels–Alder reactions include at least one heteroatom are also called a group of hetero-Diels–Alder reactions[8] Carbonyl groups, for example, can successfully react with dienes to yield dihydropyran rings, a reaction known as the oxo-Diels–Alder reaction.



Oxo- DA is a reaction of a suitable diene with an aldehyde, this reaction is important to synthetic organic chemistry.

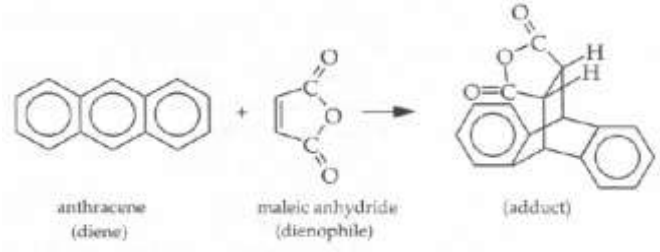
The oxo-DA reaction was first reported in 1949 using a methylpentadiene and formaldehyde as reactants [9].



To get the best acting of reactants, a chemist can control the stereochemistry [10] of a Diels-Alder product at up to four carbon atoms.

Maleic anhydride is a very good dienophile[11] because two strongly electron-with drawing groups are joined to the double bond. The diene is anthracene, which is a usual thought as an aromatic compound, and not as a diene. However, in polynuclear aromatic compounds like anthracene, each single ring may not be as well steady as a separate benzene ring.

Anthracene has only *14π* electrons, compared with 18 needed for having their own three fully aromatic rings[12].



Using an ultra- sonic technique [13, 14] and Diels-Alder reaction open a new path to improve synthesis of organic compounds.

2. Materials & Methods

(2g) of compounds [15] (naphthalene (6), naphthalene-2-ol(7), and, anthracene(8)) separately react with (1.6g) of benzaldehyde (5) for(20min), using ultrasound technique, collect the settled products (9, 10, 11) each one alone.

3. Results & Discussion

By using the two methods above, benzaldehyde was reacted with compounds (6, 7, 8) individually, using ultrasound technique instead of reflex and without catalyst as it was used in the usually preparation to obtain new hetrocyclic compounds (9, 10, 11) which are depicted in scheme (1):

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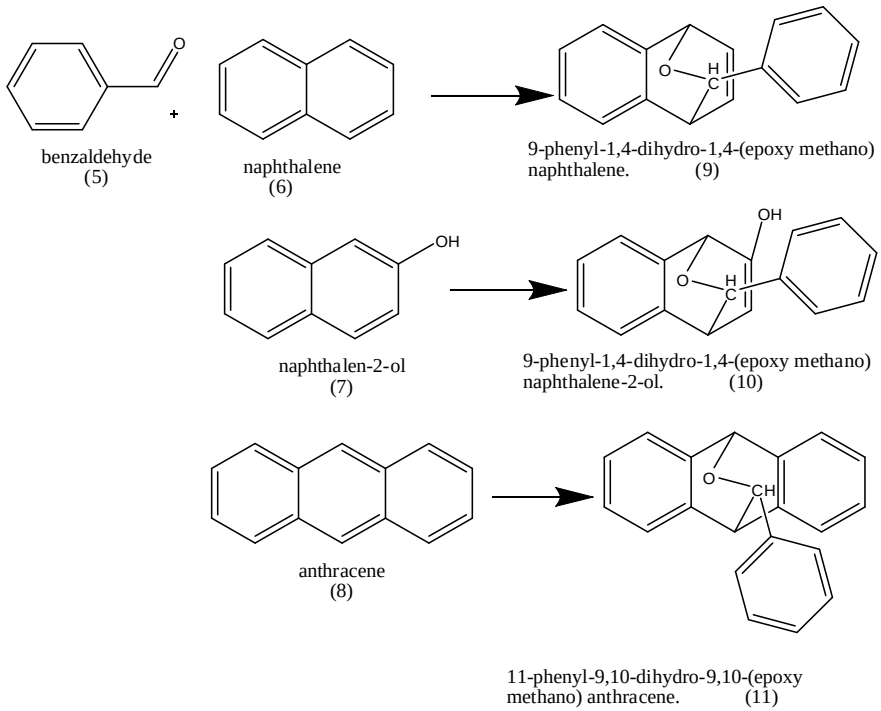

Scheme-1: path way for the prepared heterocyclic compounds.

The prepared compounds (9, 10, 11) were identified by some techniques like elemental analysis, infrared [16] and ^{1}HNMR [17].

Compounds (9, 10, 11) IR analysis showed new bands appeared for (C-O) at (1915.21cm^{-1}, 1921.13cm^{-1}, 1927.39cm^{-1}).

A band at (1716cm^{-1}) was not shown for compounds (9, 10, 11) IR analysis which is belongs to (C=O) in bezaldehyde compounds (5).
(=C-H$_{str.}$) band in compounds (9, 10, 11) appear at (3070.97cm^{-1}, 3051.65 cm^{-1}, 3047.96cm^{-1}).

For (C=C$_{str.}$) in compounds (9, 10, 11) shown band at (1686.18cm^{-1}, 1629.37cm^{-1}, 1619.49cm^{-1}).

Table (1) show M.p, R$_f$, CHNS & Yields for the prepared compounds.

Table 1: M.P *(C^o)*, R$_f$, CHNS & Yields %.

Compds.no.	*m.pC^o*	*TLC*	*CHNS*	*Yields%*
9	118-119	0.467	C=87.2, H= 5.9	28.14
10	117-119	0.747	C=81.5, H= 5.7	82.42
11	215-217	0.226	C=88.8, H= 5.6	61.74

9-phenyl-1, 4-dihydro-1, 4-(epoxy methano) naphthalene (9) :-
^{1}HNMR (300 MHz, CDCl$_3$): 4(1H, t, CH), 5.31(1H, s, CHO), ~5(1H, d, CH), 6 (1H, d, =CH), 6 (1H, d, =CH), 7.22~7.31 (2H, d, 3H, t, C$_6$H$_5$)), 7.26~7.47 (2H, d, 2H, t, C$_6$H$_4$).

9-phenyl-1, 4-dihydro-1, 4-(epoxy methano) naphthalene-2-ol (10):-^{1}HNMR (300 MHz, CDCl$_3$): 4(1H, t, CH), 5.3(1H, s, CHO), ~5(1H, d, CH), 16.77 (1H, s, OH), 6 (1H, d, =CH), 7.22~7.33 (2H, d, 3H, t, C$_6$H$_5$)), 7.25 ~7.47 (2H, d, 2H, d, C$_6$H$_4$).

11-phenyl-9, 10-dihydro-9, 10-(epoxy ethano) anthracene (11):-^{1}HNMR (300 MHz, CDCl$_3$): 4.5(1H, d, CH), 5.77(1H, s, CHO), 5.34 (1H, d, CH), 7.17~7.51 (2H, d, 3H, t, C$_6$H$_5$)), 7.17~7.51 (4H, d, 4H, t, 2C$_6$H$_4$).

4. Conclusions

Future research will involve the preparation of additional products.

This research, combined with other repetition of Diels-Alder process should provide a global library for new compounds have biological activity.

A new technique was used in this research which considered as a green chemical reaction. Using an ultra-sonic technique reduced reaction time which usually used in reflex technique in similar reactions.

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