The modern Microwave technique used to synthesis new derivatives of 1,3 – Oxazepine – 4 , 7 - dione, compounds

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Abstract

In this search, we take the direct reaction line between the aromatic aldehydes & aromatics ammines for reaction in Microwave technique to obtain clean and safe chemistry with very short time and high products, high purity comparative with the thermal method (Reflux).

The above compounds allowed to reaction to produce (hydrazon) or which named (Schiff bases) compounds (1, 3, 5, 7, 9).

The last step is cyclization step by irradiation with Microwave too, by reaction between hydrazon compounds, and phthalic anhydride to produce the (1, 3 – Oxazepine) new derivatives (2, 4, 6, 8, 10) as a final compounds, which employed in medical and pharmaceutical research's.

The prepared compounds were identified using melting point apparatus, Infrared Spectroscopy; the results are agreement with the proposed assigned to the synthesized compounds.

Key words: Microwave Technique, Hydrazon, Oxazipne.

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استخدام تقنية المايكروويف الحديثة لتحضير المشتقات الجديدة لمركبات (1 – 3 – أوكسازبين – 4 ، 7 – دايون)

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[1 ، 2] قسم الكيمياء الصيدلانية ، كلية الصيدلة ، جامعة تكريت ، نكريت ، العراق

[3] قسم الكيمياء ، كلية العلوم ، جامعة تكريت ، نكريت ، العراق

المستخلص

في هذا البحث تم اتباع خط التفاعل المباشر بين الاليدهايدات الاروماتية المعوضة ، والامينات الاروماتية المعوضة أيضاً للمفاعلة بواسطة التشعيع في جهاز المايكرووي ف الحديث لغرض الحصول على كيمياء نظيفة وامينة وبوقت زمني قصير جداً ومنتوج عالي مقارنة بالطريقة الحرارية القديمة (Reflux) ، إذ تم مفاعلة المركبات أعلاه لانتاج مشتقات قواعد شيف أو ما يسمى بمركبات الهيدروزونات الوسطية المركبات (1، 3، 5، 7، 9) والتي تم اجراء عملية الغلق الحلقي لها بخطوة تفاعل لاحقة بواسطة التشعيع أيضاً ، وذلك بمفاعلتها مع أنهيدريد الفثاليك للحصول على مشتقات (1 – 3 – أوكسازبين الجديدة) كمركبات كمركبات نهائية وهي المركبات (2، 4، 6، 8، 10) والتي تستخدم في المحال البحثي الطبي والصيدلاني .

المركبات المحضرة تم تشخيصها بواسطة تقنية الاشعة تحت الحمراء (I.R) وتقنية ايجاد درجات الانصهار لها ، وكانت الاطياف معقولة ومطابقة لما تم توقعه من تراكيبها الكيميائية .

الكلمات المفتاحية: تقنية المايكروويف ، مركبات الهيدرازون ، مركبات الاوكسازبين .

Introduction

The wave number of Microwave is about (1cm – 1m) with frequency (30 GHz – 300 Hz) [1].

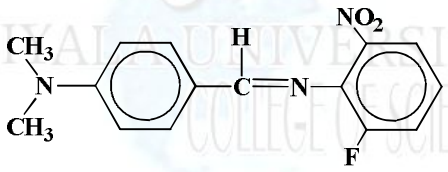
The produce heterocyclic rings by condensation cyclization reactions are type of chemicals obtained which very suitable for Microwave technique, therefor this condensation reactions by needs very high temperatures in conventional methods with special reaction conditions, like sandy or oil bathe for a long time this method temp. is about (230 – 300 °C) in oil bathe without solvent, while in modern Microwave method produce the same products with very

high yields percent, and clean chemistry with no impurities by heating for (250 ˚C) in (10) minutes [2].

The Microwave irradiation is a granted and experimental alternative method for conventional method. It very great advantages by the ability of the solid and liquid materials to transfer the electromagnetic energy through the chemical reactions heating [3]. There are number of searchers which training the Bacterial and fungal activation of hydrazon compounds and oxazipene compounds, these compounds completely like these prepared compounds in this paper. They gets this compounds are very active to the Bacteria (*Staphylo Cucsiaureus*, *Kelbesillapneumona* and *Escherichia Coli*), in addition active to the fungi (*Aspergillus, Trichophyton*) [4].

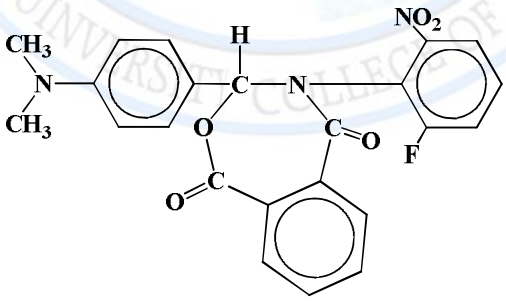
Preparation compounds by microwave (1 – 10)

1-



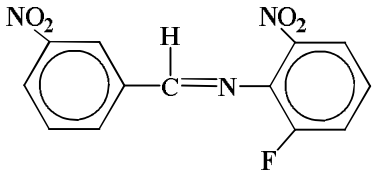
N-(4-(dimethylamino)benzylidene)-2-fluoro-6-nitroaniline

2-



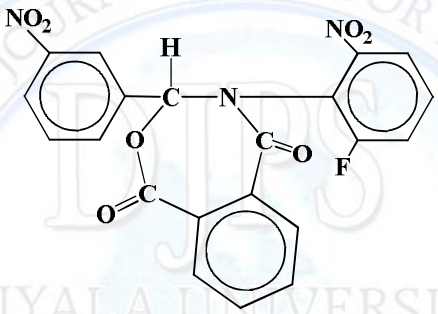
3-(4-(dimethylamino)phenyl)-4-(2-fluoro-6-nitrophenyl)-3,4-dihydrobenzo[e][1,3]oxazepine-4,7-dione

3-



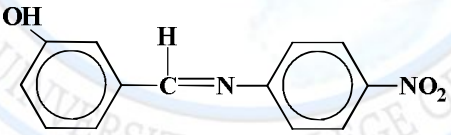
2-fluoro-6-nitro-N-(3-nitrobenzylidene)aniline

4-



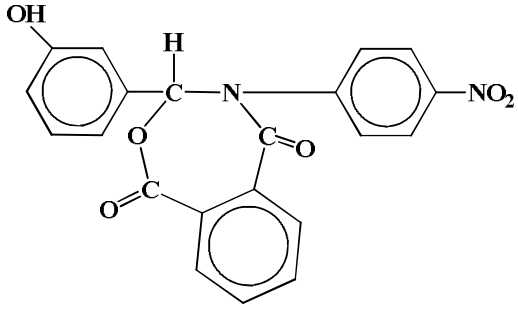
4-(2-fluoro-6-nitrophenyl)-3-(3-nitrophenyl)-3,4-dihydrobenzo[e][1,3]oxazepine-4,7-dione

5-



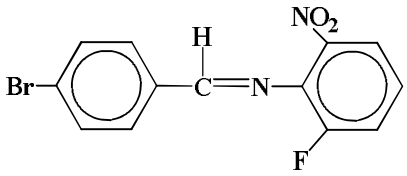
3-((4-nitrophenylimino)methyl)phenol

6-



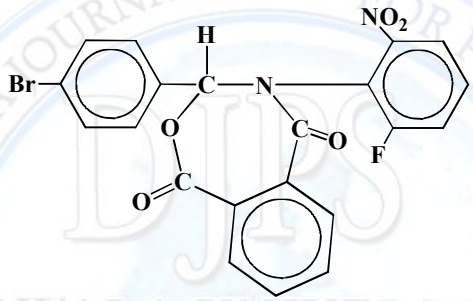
3-(3-hydroxyphenyl)-4-(4-nitrophenyl)-3,4-dihydrobenzo[e][1,3]oxazepine-4,7-dione

7-



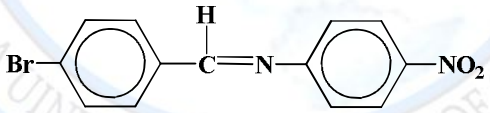
N-(4-bromobenzylidene)-2-fluoro-6-nitroaniline

8-



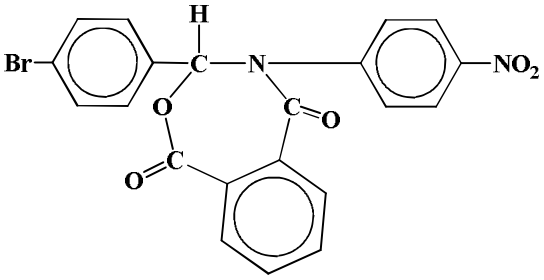
3-(4-bromophenyl)-4-(2-fluoro-6-nitrophenyl)-3,4-dihydrobenzo[e][1,3]oxazepine-4,7-dione

9-



N-(4-bromobenzylidene)-4-nitroaniline

10-



3-(4-bromophenyl)-4-(4-nitrophenyl)-3,4-dihydrobenzo[e][1,3]oxazepine-4,7-dione

Experimental part

Materials:

All materials were from Aldrich and were used further purification.

Instruments:

a. Microwellengrate 8020 (privilege).
b. FT. IR Spectrophotometer Model Shimadzu 8400.
c. Melting point apparatus Model Gallen Kamp (11Hz).

Synthesis of hydrazon (Schiff bases) Compounds [5]

Take (0.005 mol.) from aromatic aldehyds mixed and crashed carefully with (0.005 mol.) from aromatic amines, this mixture irradiated by Microwave technique for (2, 1, 2, 1.8, 1.10) minutes to give compounds (1, 3, 5, 7, 9) respectively, the products were cooled and recrystallized by absolute ethanol.

Synthesis of 1,3 – Oxazepine – 4 , 7 - dione, as final compounds [6]

A (0.005 mol.) from hydrazon compounds (Schiff bases) were mixed and crashed with (0.005 mol.) of dry phathalic anhydride, this mixture irradiated by Microwave technique for (1, 1.3, 40 sec., 40 sec., 3) minutes to yield the 1,3 – Oxazepine compounds, (2, 4, 6, 8, 10) respectively, the products were cooled and recrystallized by absolute ethanol.

Physical properties for the prepared compounds

Comp No.	Colour	Moleculer Formula	Yield %	M.P C°	M. WI Power (Waat)	React time / minute	Recrystallization Solvent
1	Yellow powder	$C_{15}H_{14}N_3O_2F$	95	76 – 78	510 w	2	Abs Ethanol
2	Orange powder	$C_{23}H_{18}N_3O_5F$	90	144 – 146	360 w	1	Abs Ethanol
3	Pall Yellow powder	$C_{34}H_8N_3O_4F$	92	158 – 160	180 w	1	Abs Ethanol
4	Dark White powder	$C_{21}H_{12}N_3O_7F$	98	147 – 149	360 w	1.3	Abs Ethanol
5	Dark Yellow powder	$C_7H_{10}N_2O_3$	96	83 – 85	360 w	2	Abs Ethanol
6	Orange powder	$C_4H_{14}N_2O_6$	95	138 – 140	360 w	40 sec	Abs Ethanol
7	White powder	$C_{13}H_8N_2O_2BrF$	97	103 – 105	360 w	2	Abs Ethanol
8	Dark White powder	$C_{21}H_{12}N_2O_5BrF$	91	76 - 78	360 w	40 sec	Abs Ethanol
9	Yellow powder	$C_{13}H_9N_2O_2Br$	90	132 – 134	510 w	1.20	Abs Ethanol
10	Yellow powder	$C_{21}H_{13}N_2O_5Br$	92	184 – 186	800 w	3	Abs Ethanol

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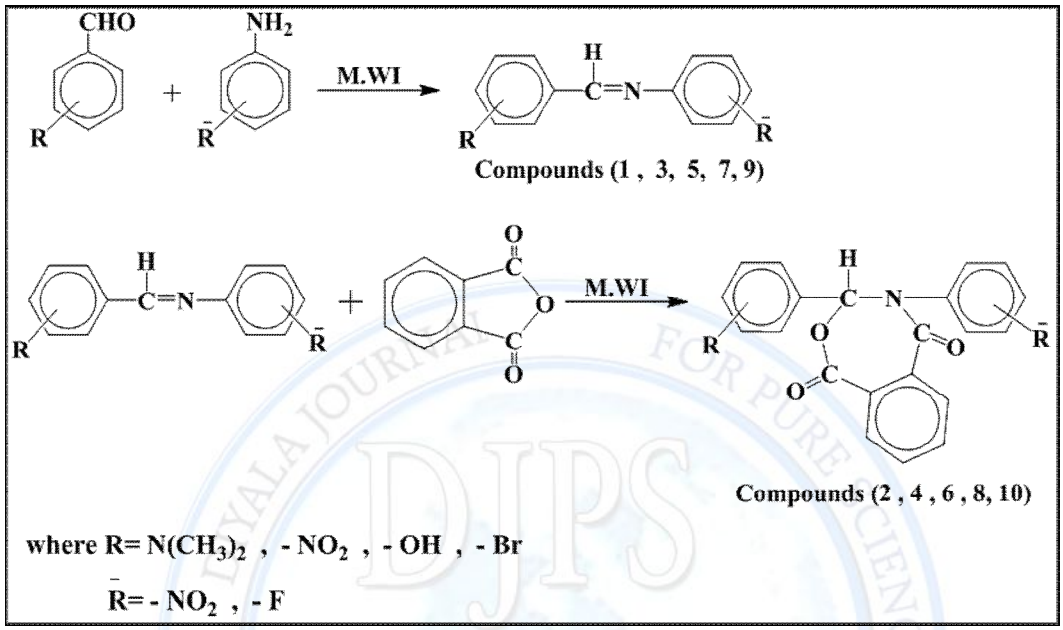
The Reaction Scheme:



Results & discussion

The modern Microwave technique used to prepares the (Schiff bases) hydrazon compounds (1, 3, 5, 7, 9). FT, I.R Spectral data showed the bands of the functional groups in the above compounds, we take the chart's for compound No. 1 as a sample for that compounds.

The bands at (1342 cm^{-1} & 1515 cm^{-1}), for – NO_2 group. The band in (1647 cm^{-1}) for $\diagup C{=}N-$ and the band at (1166 cm^{-1}) for Fluor element and the bands at (2830 cm^{-1} & 2880 cm^{-1}) for (– N – CH_3) group, and the band at (1593 cm^{-1}) for (– C = C –) group in aromatic rings.

The compounds (2, 4, 6, 8, 10) is the 1,3 – Oxazepine – 4 , 7 - dione, compounds the chart's for compounds No. 6 as a sample for them, the bands at (3201 – 3485 cm^{-1}) for – OH group, and the band at (3028 cm^{-1}) for aromatic (C – H) group, the band at (1670 cm^{-1}) for lactam $-\overset{|}{N}-\overset{O}{\overset{\|}{C}}-$ group, the bands at (1340 & 1500 cm^{-1}) for – NO_2 group, the band at (1145 cm^{-1}) for (– O – C) Ether group.

Finally the band at (1589 cm^{-1}) for the aromatic (– C = C –) group [7].

References

1. Rassayan J. of chemistry, Vol. I, No. 1. Pp 68 – 92 (2008).
2. W. Stadlbauer, O. Schmat, T.Kappe, Monatsh, Chem. 111, Pp 105 – 113, (1980).
3. K. Mohammed, Ziallha, Mobeen Rany and H.E.J. Research. Institute of Chemistry international center for chemical Sciences University of Karachi, letters in Organic Chemistry, 1, 50 – 52, (2004).
4. Abed M. Daher Al-Joubory. Ph.D thesis "Synthesis and Comparative Study of some Heterocyclic Compounds Containing Five and Seven – Membered Rings By Conventional and Microwave Method (MAOS) and Evaluate the Biological activity of Some of Them", Tikrit University , Education College Pp. 103 – 105, (2010).
5. A. Cansiz, M. Koparir and A. Demirdag Molecules, 9, 204 (2004).
6. Firas Shawki Abdul-Razzak. AL-Joboury Ph. D. synthesis of New Spiro Fused ring Ibn Al-Haitham Education College Baghdad University p. 81(2006).
7. Williams and Fleming, Spectroscopic methods inorganic chemistry. 2nd. Ed. Cambridge College U.K. (1973).

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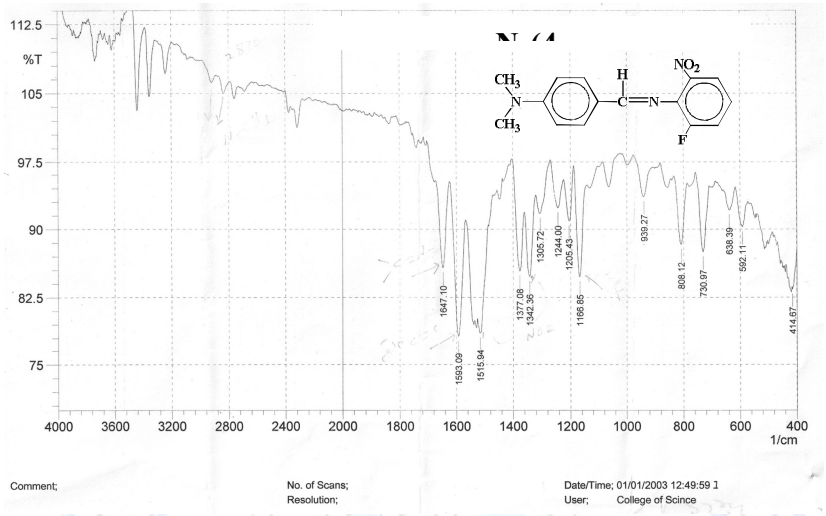

Compound No. (1):