Microwave Synthesis of some new compounds
1,3 – Oxazepine – 4 , 7 - dione,derivatives from Schiff bases

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Abstract:
The Microwave technique is a new method for synthesis of organic compounds, which take a pure and high percent of products and completed with short time, compared with conventional method (heating method). The prepared Schiff bases allowed to react by Microwave technique which is a new apparatus for synthesis the organic compounds, reacts with Malic anhydride to yield (1, 3, 6, 7) 1,3 – Oxazepine compounds and reacted with phthalic anhydrides to yield (2, 4, 5, 8) 1,3 – Oxazepine compounds. The useful thing in Microwave technique is using without solvent there for it called (dry method). The synthesized compounds were identified using Melting point apparatus, Infrared Spectroscopy.

Keywords : Microwave Synthesis , 1,3 – Oxazepine – 4 , 7 - dione,derivatives from Schiff bases

Introduction:
Microwave technique method is appears lastly which employed wildly as a method instead of the (Reflexing method) in organic synthesis, because this method is effective and economic, there for it called (MAOS) method, by this method we have high percent and high purity of product compounds with chosen reactions, and the reacts completed with very short time compared with conventional methods.

Microwave method acts by mechanical (Ionic motion) which generated by the passed electrical field in chemical reaction. When we increase the power of Microwave radiation the heat of the perimeter of reaction increase too, these for the conductance energies more effective especially when the reaction is Ionic material this cause absorption for the Microwave rapidly, and cause chemical reaction soon

Oxazepine compounds are seven – membered heterocyclic unsaturated ring. This ring contain five carbon atoms and one oxygen atom, and one nitrogen atom too, there are three isomers for Oxazepine compounds 1, 2 and 1, 3 and 1, 4 – Oxazepine.

There are different ways for prepare Oxazepine seven – membered heterocyclic ring, for example direct addition of malic anhydride and phthalic anhydride and succinic anhydride to double bond

\[
\text{C} = \text{N} -
\]

for Schiff bases.

The Microwave is an electromagnetic radiation with frequency about (0.3 to 300 GHz).

The principle of Microwave in chemical reactions it is by heating the chemical material with effect of (thermal Microwave constant) this phenomena depend on specific ability for material to absorption of Microwave energy, which by this (heating) can be converts to a new chemical material and new bonds between atoms.
Synthesized Compounds (1 – 8)

1-

\[
\text{O}_2\text{N} - \text{N} = \text{N} - \text{N} = \text{N} - \text{C} = \text{O}
\]

2-(4-bromo-phenyl)-3-(4-nitrophenyl)-1,2-dihydro-1,3-oxazepine-4,7-dione

2-

\[
\text{Br} - \text{N} - \text{C} = \text{O} - \text{N} = \text{C} = \text{O}
\]

3-(4-bromo-phenyl)-1,1,5-dimethyl-3-oxa-2-phenyl-2-oxazolidin-4-yn-3,4-dihydro-1,3-oxazepine-4,7-dione

3-

\[
\text{O} - \text{C} - \text{N} = \text{C} = \text{O} - \text{N} = \text{N} - \text{N} = \text{N} - \text{N} = \text{H}
\]

3,3’-(4,4’-biphenyl)-4,4’-diyl][bis(diazene-2,1-diyl)][bis(4,1-phenylene)]

bis(2-(4-hydroxyphenyl)-2,3-dihydro-1,3-oxazepine-4,7-dione)

4-

\[
\text{NO}_2 - \text{N} = \text{N} - \text{N} - \text{C} = \text{O}
\]

4,4’-bicyclo[2,4,0]octane-1,6-diyl][bis(2,3-dihydro-5,5-dimethyl-1H-indazole-1,3-oxazepine-4,7-dione)
Experimental:
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 (11H).

Synthesis of the 1,3 – Oxazepine compounds
a. Take (0.01 mol) from prepared subalial Schiff base, mixed and crashed with (0.01 mol) of dry Malic anhydride and irradiated by Microwave technique for (6, 5, 6, 5) minutes yield the compounds (1, 3, 6, 7) respectively, the products cool and recrystallized by ethanol.

b. Take (0.01 mol) from prepared subalial Schiff base, mixed and crashed with (0.01 mol) of dry phthalic anhydride and and irradiated by Microwave technique for (20, 3, 10, 5) minutes yield the compounds (2, 4, 5, 8) respectively, the products cool and recrystallized by ethanol.
Physical properties for the prepared compounds

<table>
<thead>
<tr>
<th>Comp No.</th>
<th>Schiff bases parent starting material M.P (°C)</th>
<th>Colour</th>
<th>Molecule Formula</th>
<th>Yield %</th>
<th>M.P (°C)</th>
<th>M. Wt Power (Watt)</th>
<th>React time / minute</th>
<th>Recrystlization Solvent</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>138 – 140 Yellow powder C\textsubscript{23}H\textsubscript{12}N\textsubscript{4}O\textsubscript{2}Br</td>
<td>85</td>
<td>160 – 162</td>
<td>6</td>
<td>Abs</td>
<td>Ethanol</td>
<td></td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>254 – 259 Yellow powder C\textsubscript{25}H\textsubscript{13}N\textsubscript{4}O\textsubscript{4}Br</td>
<td>90</td>
<td>196 – 198</td>
<td>20</td>
<td>Abs</td>
<td>Ethanol</td>
<td></td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>140 – 144 Black powder C\textsubscript{16}H\textsubscript{10}N\textsubscript{2}O\textsubscript{2}</td>
<td>86</td>
<td>175 – 180</td>
<td>5</td>
<td>Abs</td>
<td>Ethanol</td>
<td></td>
<td></td>
</tr>
<tr>
<td>4</td>
<td>128 – 130 Brown powder C\textsubscript{6}H\textsubscript{12}N\textsubscript{4}O\textsubscript{12}</td>
<td>75</td>
<td>255 – 259</td>
<td>3</td>
<td>Abs</td>
<td>Ethanol</td>
<td></td>
<td></td>
</tr>
<tr>
<td>5</td>
<td>220 – 223 Yellow powder C\textsubscript{2}H\textsubscript{2}N\textsubscript{2}O\textsubscript{4}</td>
<td>89</td>
<td>284 – 286</td>
<td>10</td>
<td>Abs</td>
<td>Ethanol</td>
<td></td>
<td></td>
</tr>
<tr>
<td>6</td>
<td>220 – 222 White powder C\textsubscript{2}H\textsubscript{2}N\textsubscript{2}O\textsubscript{4}</td>
<td>95</td>
<td>225 – 228</td>
<td>6</td>
<td>Abs</td>
<td>Ethanol</td>
<td></td>
<td></td>
</tr>
<tr>
<td>7</td>
<td>130 – 135 Orange powder C\textsubscript{32}H\textsubscript{2}N\textsubscript{2}O\textsubscript{2}S\textsubscript{2}</td>
<td>80</td>
<td>181 – 184</td>
<td>5</td>
<td>Abs</td>
<td>Ethanol</td>
<td></td>
<td></td>
</tr>
<tr>
<td>8</td>
<td>130 – 135 Yellow powder C\textsubscript{4}H\textsubscript{2}N\textsubscript{2}O\textsubscript{2}S\textsubscript{2}</td>
<td>50</td>
<td>164 - 168</td>
<td>5</td>
<td>Abs</td>
<td>Ethanol</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

The reaction scheme:

Result and discussion

The synthesis compounds by (Microwave technique) started from the aromatic Schiff bases to produce the (1 – 8) 1,3 – Oxazepine -4, 7 dione compounds and 1,3 – Oxazepine -4, 5- dione. FT. IR. Spectral data showed the bands of the functional groups that substituted in rings or in others. Because the more resembles between the compounds, we take the compounds (4) & (7) as a sample. The bands at 1585 cm\textsuperscript{-1} for azo. groups (\(\text{--)N=N(--)}\) in (1, 3, 4) compounds.

The observed bands at (1338 & 1404) cm\textsuperscript{-1} for (\(\text{--)N=O(--)}\) groups. The lactam (\(\text{--)C=N(--)}\) bands observed at 1691 cm\textsuperscript{-1}, and the phenol (\(\text{--)OH(--)}\) group observed at 3456 cm\textsuperscript{-1}, the thiourea (\(\text{--)S=H(--)}\) bands observed at 259 cm\textsuperscript{-1}, the aromatic (\(\text{--)C=C(--)}\) bands observed at (159 –1600) cm\textsuperscript{-1}, the mono (\(\text{--)C=H(--)}\) substitution on the phenyl rings in compound (7) bands observed at 707 cm\textsuperscript{-1}.

Finally the bands of (C-H) aryl and (C-H) aliphatic observed at 3190 cm\textsuperscript{-1} and 2958 cm\textsuperscript{-1} respectively [7].
References:


تحضير بعض المشتقات الجديدة لمركبات 1،3،6،7،4،أوكسازبين بواسطة تقنية المايكرووية

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الخلاصة

أن تقنية المايكرووية هي تقنية جديدة على الساحة البحثية العلمية وخاصة في تحضير المركبات العضوية، ويمكن بواسطة هذه التقنية الحصول على نسبة عالية وثيقة من المنتج بالإضافة إلى وقت قصير جداً مقارنة بالطريقة القديمة التي كانت تستخدم في تحضير العضوي تم مفاصله قواعد شيف المحضرة سابقاً، والتي استخدمت في تحضير المركبات العضوية حديثاً إذ تم مفاعاتها مع إنهدريد الماليك لإنتاج مشتقات (1،3،6،7،4،أوكسازبين – 4،1،3،6،7،4،أوكسازبين – 4،1،3،6،7،4،أوكسازبين – 4،7 - دايون الجديد أيضاً . والمواد في تقنية المايكرووية أنها تم بدون استخدام المذيب ولذا فهي تسمى بالطريقة الجافة. المركبات المحضرة تم تشخيصها بواسطة طيف (I.R) الأشعة تحت الحمراء، ودرجة الاصهار .