SYNTHESIS AND CHARACTERIZATION OF SOME BROMO SUBSTITUTED CHALCONE BY THE GREEN SYNTHESIS WAY (GRINDING METHOD) AND AURONES 2-BENZYLIDINE-1-BENZOFURAN-3-ONE BY CYCLIZATION METHOD.

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ABSTRACT
The presented aurones were synthesized by cyclization of chalcones which are prepared from 2- hydroxy 5- bromo acetophenone and different aldehydes a series of bromo substituted aurones were synthesized and characterized through NMR, IR, and elemental analysis. The synthetic methods used proved successful synthesis of the desired compound. Grinding technique is used to follow the green synthesis way for the synthesis of chalcone.

KEYWORDS: Chalcones, Aurones, Green Synthesis (Grinding Technique).

INTRODUCTION
Aurones are the important type of flavanoid, responsible for the bright yellow colour of flower. Previous studies have shown that aurones exhibit anti-cancer activity as well as a variety of other pharmacological activities, including anti-inflammatory and anti-viral properties. More specifically, these studies indicate that aurones show potential in inhibiting cyclooxygenase-2 activity (COX-2), which plays an integral part of inflammation and its associated diseases, such as cancer. Furthermore, structure-activity studies have shown that halogen substituents increase anti-cancer activities of flavonoid derivatives. Therefore, the evaluation of the “A” ring halogen substituted aurone is of great importance.
EXPERIMENTAL SECTION
SYNTHESIS OF CHALCONE
The chalcones were synthesized by using 5-bromo 2-hydroxy acetophenone with some aldehydes (anisaldehyde, furfuraldehyde, bezaldehyde, cinnamaldehyde, salysilaldehyde) under alkaline condition. The chalcones were obtained yellow to brown in color their synthesis were confirm by melting point method and from spectral interpretation. This is the general claisen Schmidt reaction[23] but I use the reaction by grinding was carried out in a mortar and pestle at room temperature. During the grinding, sudden change in color took place, indicating the progress of reaction. The full conversion of the reaction mixture to a solid mass indicates the completion of reaction.

General procedure
A mixture of 5-bromo 2-hydroxy acetophenone (4 mmol), aryl aldehyde (4.1 mmol) and ammonium bromide and ammonium persulphate moist with few drops of water[25] is ground at room temperature for 25 minutes in a mortar and pestle. The reaction mixture is allowed to stand for 25 min. Then Ice-cold water (30 ml) was added to the reaction mixture and acidified with conc. HCl. The product was collected by vacuum filtration and then recrystallized from ethanol. The same procedure of grinding was apply by using anhydrous bariun hydroxide and then result of two procedure was compaired.

RESULTS AND DISCUSSION
Chalcone of Benzaldehyde with ketone (C1)

Chalcone of cinnamaldehyde with ketone (C2)
Chalcones of furfuraldehyde with ketone (C3)

\[
\text{Br} \quad \text{OH} \\
\text{CH}_3 
\]

\[\text{Br} \quad \text{OH} \quad + \quad \text{Ba(OH)}_2 \quad \text{or} \quad \text{NH}_4\text{Br}(\text{NH}_4)_2\text{S}_2\text{O}_8 \quad \rightarrow \quad \text{Br} \quad \text{OH} \quad \text{CH}_3 
\]

Chalcone of salisylaldehyde with ketone (C4)

\[
\text{Br} \quad \text{O} \quad \text{CH}_3 
\]

\[\text{Br} \quad \text{O} \quad \text{CHO} \quad + \quad \text{Ba(OH)}_2 \quad \text{or} \quad \text{NH}_4\text{Br}(\text{NH}_4)_2\text{S}_2\text{O}_8 \quad \rightarrow \quad \text{Br} \quad \text{O} \quad \text{CH}_3 
\]

Chalcone of anisaldehyde with ketone (C5)

\[
\text{Br} \quad \text{O} \quad \text{CH}_3 
\]

\[\text{Br} \quad \text{O} \quad \text{CHO} \quad + \quad \text{Ba(OH)}_2 \quad \text{or} \quad \text{NH}_4\text{Br}(\text{NH}_4)_2\text{S}_2\text{O}_8 \quad \rightarrow \quad \text{Br} \quad \text{O} \quad \text{CH}_3 
\]

The time duration for the formation of chalcone taken by both reagent was near about same but the yield obtained from barium hydroxide was superior than the ammonium bromide and ammonium persulphate.

Synthesis of Aurones from chalcones: about 3.35 gm (0.01 M) of chalcones , 2.35 gm of (0.01M) mercuric chloride , 20 ml of DMSO in round bottom flask. Reflux the reaction mixture for 3 hours, then reaction mixture was hydrolysed by using acidified ice cold water, filter the crude product and wash it 3-4 times by distilled water, dried, and crystallized by ethanol, solid product was obtained i.e aurone.

Auone of Anisaldehyde (A1)
Aurone of benzaldehyde (A2)

Aurone of cinnamaldehyde (A3)

Aurone of furfuraldehyde (A4)

Aurone of salisylaldehyde (A5)

Instrumentation

Table 1: Physical data of chalcones (melting point & colour)

<table>
<thead>
<tr>
<th>Sr.NO</th>
<th>Symbol</th>
<th>Name of compound</th>
<th>Melting point</th>
<th>Colour</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>C1</td>
<td>5-bromo2-hydroxy(1,3)diphenyl-2 propen-1 one</td>
<td>81°C</td>
<td>Brown</td>
</tr>
<tr>
<td>2</td>
<td>C2</td>
<td>5-bromo2-hydroxy(1,5)diphenyl-2,4 -butadien-1 one</td>
<td>80°C</td>
<td>yellow</td>
</tr>
<tr>
<td>3</td>
<td>C3</td>
<td>5-bromo2-hydroxy-3furan 1-phenyl -2 propen-1 one</td>
<td>79°C</td>
<td>Yellowish brown</td>
</tr>
<tr>
<td>4</td>
<td>C4</td>
<td>5-bromo2-hydroxy 4 hydroxy (1,3)diphenyl-2 propen-1 one</td>
<td>129°C</td>
<td>Greenish yellow</td>
</tr>
<tr>
<td>5</td>
<td>C5</td>
<td>5-bromo2-hydroxy 4- methoxy (1,3)diphenyl-2 propen-1 one</td>
<td>82°C</td>
<td>Golden yellow</td>
</tr>
</tbody>
</table>
Table 2: Physical data of substituted benzofuran i.e. aurone (melting point & colour)

<table>
<thead>
<tr>
<th>Sr.no</th>
<th>Symbol</th>
<th>Name</th>
<th>Melting point</th>
<th>Colour</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>A1</td>
<td>5- bromo 4- methoxy 2-Benzylidene-1-benzofuran-3-one</td>
<td>89 °c</td>
<td>Greenish yellow</td>
</tr>
<tr>
<td>2</td>
<td>A2</td>
<td>5- bromo 2-Benzylidene-1-benzofuran-3-one</td>
<td>95 °c</td>
<td>Greenish yellow</td>
</tr>
<tr>
<td>3</td>
<td>A3</td>
<td>5- bromo(1,3)butadiene-3-Benzylidene-1-benzofuran-3-one</td>
<td>88 °c</td>
<td>Golden yellow</td>
</tr>
<tr>
<td>4</td>
<td>A4</td>
<td>5- bromo 2-furane-1-benzofuran-3-one</td>
<td>102 °c</td>
<td>yellow</td>
</tr>
<tr>
<td>5</td>
<td>A5</td>
<td>5- bromo 4-hydroxy-2-Benzylidene-1-benzofuran-3-one</td>
<td>85 °c</td>
<td>Yellowish brown</td>
</tr>
</tbody>
</table>

Infra-red spectroscopy

Table 3: The IR spectral analysis of compound showed the presence of following absorption bands

<table>
<thead>
<tr>
<th>Sr. no</th>
<th>Name of compound</th>
<th>V(C=O) cm⁻¹</th>
<th>V(c-o) cm⁻¹</th>
<th>V(c=c) cm⁻¹</th>
<th>V(-Br) cm⁻¹</th>
<th>Any special substituent</th>
<th>V(c=c) cm⁻¹</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Cyclic</td>
<td>aliphatic</td>
<td>Para</td>
<td>substituted</td>
<td>Aromatic</td>
<td></td>
</tr>
<tr>
<td>1</td>
<td>A-1</td>
<td>1692</td>
<td>1302</td>
<td>1636</td>
<td>694</td>
<td>V –OCH₃ 2835</td>
<td>1562</td>
</tr>
<tr>
<td>2</td>
<td>A-2</td>
<td>1681</td>
<td>1274</td>
<td>1640</td>
<td>700</td>
<td>-</td>
<td>1596</td>
</tr>
<tr>
<td>3</td>
<td>A-3</td>
<td>1694</td>
<td>1216</td>
<td>1636</td>
<td>684</td>
<td>1636 conjugated diene</td>
<td>1556</td>
</tr>
<tr>
<td>4</td>
<td>A-4</td>
<td>1690</td>
<td>1019</td>
<td>1641</td>
<td>628</td>
<td>1208 furan ring –O-</td>
<td>1579</td>
</tr>
<tr>
<td>5</td>
<td>A-5</td>
<td>1636</td>
<td>1306</td>
<td>1670</td>
<td>664</td>
<td>3236 (-OH)</td>
<td>1598</td>
</tr>
</tbody>
</table>

Table 4: The H¹ NMR spectral analysis of compound showed the presence of following absorption bands

<table>
<thead>
<tr>
<th>Sr. no</th>
<th>Name of compound</th>
<th>(δ ppm)</th>
<th>No.of potons</th>
<th>assingment</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>3.33, 7.6</td>
<td>3H ,1H</td>
<td>Ar-O-CH₃, Ar-H</td>
</tr>
<tr>
<td>1</td>
<td>A-1</td>
<td>7.84, 3.8</td>
<td>1H,1H,1H</td>
<td>Ar-H, C=CH</td>
</tr>
<tr>
<td>2</td>
<td>A-2</td>
<td>7.13, 2.4, 8.17</td>
<td>1H, 1H, 1H,1H</td>
<td>Ar-H, C=CH</td>
</tr>
<tr>
<td>3</td>
<td>A-3</td>
<td>7.16, 2.5</td>
<td>1H, 1H, 1H</td>
<td>Ar-H, C=CH</td>
</tr>
<tr>
<td>4</td>
<td>A-4</td>
<td>6.64, 3.326</td>
<td>1H, 1H, 1H,1H</td>
<td>Ar-H, C=CH</td>
</tr>
<tr>
<td>5</td>
<td>A-5</td>
<td>5.80, 7.44, 3.32</td>
<td>1H, 1H, 1H,1H</td>
<td>Ar-O-H, Ar-H, C=CH</td>
</tr>
</tbody>
</table>


As the aurones are responsible for the bright yellow colour of flower thus attempt has been made to see the effect on the flower of white colour is they show colour change . for this Catharanthus roseus plant was selected but no significant change of colour was obtained .

CONCLUSION
The compound i.e substituted benzofuran was succesfully synthesized by the green synthesis way , the yield obtained was good their purity and conformation was checked by melting point and from spectral data .

ACKNOWLEDGEMENTS
I hearby very thankful to my friend Mr. swapnil k. warkhade who help me for this work.

REFERENCES
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