Synthesis of Chalcones by grindstone chemistry as an intermediate in Organic Synthesis

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Abstract

A facile synthesis of some novel 4-(Sub Phenyl)-1-phenyl 2-propen-1-ones (I-IX) has been achieved by the condensation of Aromatic and aliphatic ketone with various substituted aromatic aldehydes through intramolecular aldol condensation using solid NaOH by using grinding technique. All synthesized compounds were characterized on the basis of IR, NMR and UV spectroscopic data and elemental analysis.

Keywords: Chalcones, Aldol condensation, Grindstone Chemistry

Introduction

The chalcones and their derivatives are important intermediates in organic synthesis. They serve as starting material for the synthesis of variety of heterocyclic compounds which are of physiological importance. Due to the presence of enone functionality in chalcone moiety confers wide range of biological applications such as antibacterial (Opletalova, 2000), antifungal (Go et al., 2005), antitumor (Ducki et al., 1998), antiulser (Kyogoku et al., 1979), anticancer (Konieczny et al., 2007), analgesic (Viana et al., 2003), antiinflammatory (Jin et al., 2007), antileishmanial (Narender et al., 2005) etc. The utility of chalcones due to their usefulness as in synthesis of various heterocyclic compounds. Solid-state reaction occurs more efficiently and more selectively than does the solution reaction, since molecules in the crystal are arranged tightly and regularly. Reaction are simple to handle, reduce pollution, comparatively cheaper to operate and may be regarded as more economical and ecologically favorable procedure in chemistry. In the present investigation, grindstone technique was used for the synthesis of titled compounds. All these facts make them desired synthetic targets and more importantly they have been extensively employed as intermediates in the synthesis of heterocyclic and carbocyclic systems as per Scheme-II.

Materials and Methods

All chemicals were of synthetic grade (S. D. Fine Chem. Ltd. Mumbai, India). The melting points were recorded on electro-thermal apparatus and are uncorrected.

The purity of the compounds was checked by TLC on precoated SiO$_2$ gel (HF254, 200 mesh) aluminium plates (E Merk) using hexane and ethyl acetate visualized in iodine chamber. IR spectra were recorded in KBr on a perkin-Elmer model-983. 1HNMR spectrum recorded on Varian Mercury 300 MHz instrument using CDCl$_3$, DMSO-d6 as solvent (chemical shift in $\delta$ ppm), using TMS as internal standard. UV spectra were recorded in ethanol on Beckmann DK-1 spectrophotometer. Elemental analysis was performed on a Heraeus CHN analyzer and was within the $\pm$0.5% of the theoretical values.

Synthesis of 4-(Sub Phenyl)-1-phenyl 2-propen-1-one ($1_{(x)}$)

Different aldehydes (0.05 mmol), substituted acetophenone (0.05 mmol) and 2.0 g solid NaOH is taken in a small mortar. Grinded the mixture thoroughly for 5-10
(i) Hydrazine/EtOH (ii) NH₂NH₂/GAA (iii) O=C(NH₂)₂/EtOH/NaOH
(iv) S=C(NH₂)₂/EtOH/NaOH (v) NH₂OH.HCl,EtOH (vi) O=C(NH₂)₂
(vii) Ph.NH₂NH₂/GAA

Table 1. Physical data of synthesized compounds (I-IX)
Fig. 1. The color and nature of synthesized compounds (I-IX)

<table>
<thead>
<tr>
<th>Compound No.</th>
<th>Spectral analysis</th>
<th>Elemental analysis (Calc./Found) %</th>
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<tbody>
<tr>
<td>I</td>
<td>UV (λmax): 312 nm; IR (KBr), Vmax: -OH(3465), &gt;C=O(1658), &gt;C=C(1622) cm⁻¹; ¹H NMR (CDCl₃), δ: 7.88-7.81 (1H, d, =CH-Ar), 7.72-7.30 (8H, m, Ar-H), 6.81 (1H, d, -CO-CH=), 4.9 ppm</td>
<td>75.00 (74.92)</td>
</tr>
<tr>
<td>II</td>
<td>UV (λmax): 306 nm; IR (KBr), Vmax: &gt;C=O(1660), &gt;C=C&lt;1638 cm⁻¹; ¹H NMR (CDCl₃), δ: 7.88-7.71 (1H, d, =CH-Ar), 7.72-7.30 (10H, m, Ar-H), 6.81 (1H, d, -CO-CH=) ppm</td>
<td>86.53 (86.50)</td>
</tr>
<tr>
<td>III</td>
<td>UV (λmax): 312 nm; IR (KBr), Vmax: -OH(3465), &gt;C=O(1662), &gt;C=C&lt;1622 cm⁻¹; ¹H NMR (CDCl₃), δ: 7.88-7.81 (1H, d, =CH-Ar), 7.72-7.30 (9H, m, Ar-H), 6.81 (1H, d, -CO-CH=), 4.89 (1H, s, C-4'-OH) ppm</td>
<td>80.36 (80.35)</td>
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<tr>
<td>IV</td>
<td>UV (λmax): 308 nm; IR (KBr), Vmax: &gt;C=O(1665), &gt;C=C&lt;1620 cm⁻¹; ¹H NMR (CDCl₃), δ: 7.88-7.81 (1H, d, =CH-Ar), 7.72-7.30 (8H, m, Ar-H), 6.81 (1H, d, -CO-CH=), 5.09 (1H, s, C-2'-OH), 3.54 (3H, s, -OCH₃), 2.54 (3H, s, -COCH₃) ppm</td>
<td>75.59 (75.60)</td>
</tr>
<tr>
<td>V</td>
<td>UV (λmax): 339 nm; IR (KBr), Vmax: &gt;C=O(1678), &gt;C=C&lt;1622 cm⁻¹; ¹H NMR (CDCl₃), δ: 7.88-7.81 (1H, d, =CH-Ar), 7.85-7.30 (4H, m, Ar-H), 6.81 (1H, d, -CO-CH=), 3.83 (3H, s, -OCH₃), 2.54 (3H, s, -COCH₃) ppm</td>
<td>75.00 (75.00)</td>
</tr>
<tr>
<td>VI</td>
<td>UV (λmax): 308 nm; IR (KBr), Vmax: -OH(3465), &gt;C=O(1660), &gt;C=C&lt;1622 cm⁻¹; ¹H NMR (CDCl₃), δ: 7.88-7.81 (1H, d, =CH-Ar), 7.58-7.30 (4H, m, Ar-H), 6.81 (1H, d, -CO-CH=), 3.26 (6H, s, -NCH₃), 2.54 (3H, s, -COCH₃) ppm</td>
<td>76.19 (76.13)</td>
</tr>
</tbody>
</table>

Table 2. Spectral and elemental analysis of synthesized compounds (I-IX)
UV (λmax): 313 nm; IR (KBr), νmax: >C=O(1660), >C=C<(1622), C–O (1280) cm–1. H NMR (CDCl3), δ: 7.88–7.81 (1H, d, =CH–Ar), 7.72–7.30 (3H, m, Ar–H), 6.81 (1H, d, -CO-CH=), 4.9 (1H, s, C–4'–OH), 2.43 (3H, s, OCH3) ppm.

UV (λmax): 313 nm; IR (KBr), νmax: >C=O(1659), >C=C<(1622), C–O (1275) cm–1. H NMR (CDCl3), δ: 7.88–7.81 (1H, d, =CH–Ar), 7.72–7.30 (9H, m, Ar–H), 6.81 (1H, d, -CO-CH=), 2.83 (3H, s, OCH3) ppm.

Results and Discussion

We have developed an efficient, operationally simple, environmental benign method under solvent free condition (solid NaOH) by using grindstone chemistry. Many researches have been done on synthesis of Chalcone by using different methods such as Conventional and nonconventional methods which requires larger time and less atom economy. But our method is differing from all these methods as it requires shorter reaction time and high yields up to 75 to 85%. Chalcone is a versatile molecule which having wide range of biological and pharmacological applications as described. Due to its utility we have synthesized chalcones by grinding technique which is very fertile and easiest to all researchers. Main object in synthesis of chalcone is that it can be further used in synthesis of heterocyclic compounds i.e. it acts as an intermediates as per Scheme-II further we have examined the synthesized compounds by using analytical techniques as per Table-I and II. The nature and color of corresponding compounds as shown below:

Conclusion

We have developed a simple, efficient and more eco-friendly method for synthesis of chalcones under solvent free condition by grinding technique. The notable advantages of present method are no organic solvent required (except for the product recrystallisation), waste minimization, simple operation, clean reaction profile, easy work-up, shorter reaction time (4-8 min), high yields (75-85%) and eco-friendly as compared to conventional method.

Acknowledgement

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References


Synthesis of isomeric, oxathiolone fused chalcones and comparison, their activity towards various microorganisms and human cancer cell lines. Chemical and Pharmaceutical Bulletin 55: 817-820.


