

## Amberlyst-15 catalysed microwave assisted cross-aldol condensation between ketones and aldehydes under solvent free condition

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**Abstract :** Amberlyst-15 has been applied as an efficient heterogeneous catalyst for the first time for rapid synthesis of  $\alpha, \alpha'$ -bis(arylmethylene)cycloalkanones,  $\alpha, \alpha'$ -bis(cinnamylidene)cycloalkanones,  $\alpha$ -cinnamylideneacetophenones and chalcones in very good yield by the reaction between various aldehydes and ketones under microwave irradiation. The new process for the cross-aldol condensation reaction works well in absence of any solvent. The yields are high and the process is environmentally benign.

**Keywords :** Cycloalkanones, benzaldehydes, cinnamaldehyde, amberlyst-15, microwave irradiation,  $\alpha, \alpha'$ -bis(arylmethylene)-cycloalkanones,  $\alpha, \alpha'$ -bis(cinnamylidene)cycloalkanones,  $\alpha$ -cinnamylideneacetophenones, chalcones.

### Introduction

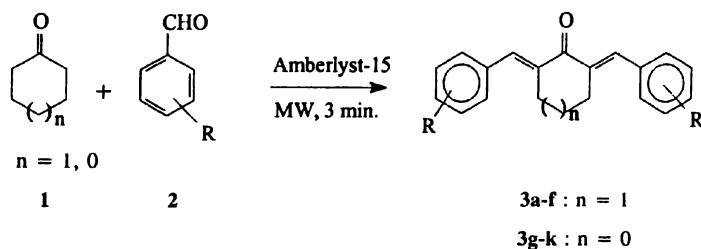
$\alpha, \alpha'$ -Bis(arylmethylene)cycloalkanones, chalcones and related compounds are used as starting materials for synthesis of a variety of other interesting compounds. Some of these starting materials as well as many of the products derived from them are known to show diverse biological activities<sup>1</sup>. Recently, we have carried out syntheses of *E*-8-(arylmethylene)-4-aryl-1,2,3,4,5,6,7,8-octahydrobenzo-[*d*]pyrimidine-2-thiones<sup>2</sup> and *E*-2-amino-4-aryl-8-(arylmethylene)-5,6,7,8-tetrahydrobenzo[*d*]pyrimidines and their lower analogues<sup>3</sup> by base-catalysed cyclocondensation of  $\alpha, \alpha'$ -bis(arylmethylene)cycloalkanones with thiourea and guanidine hydrochloride, respectively. The preparation of  $\alpha, \alpha'$ -bis(arylmethylene)cycloalkanones is usually achieved by the traditional method involving cross-aldol condensation of cycloalkanones and aromatic aldehydes in aqueous ethanolic alkali. However, survey of the literature shows that there is a tendency to develop methodologies for synthesis of such compounds by use of solid base<sup>4</sup>,  $\text{BF}_3 \cdot \text{OEt}_2$  (Ref. 5),  $\text{InCl}_3$  (Ref. 6),  $\text{InCl}_3/\text{TMSCl}$ <sup>7</sup>, bis(*p*-ethoxyphenyl)telluroxide (BOMPTO)<sup>8</sup>,  $\text{TMSCl}/\text{NaI}$ <sup>9</sup>,  $\text{Yb}(\text{OTf})_3$  (Ref. 10),  $\text{Cu}(\text{OTf})_2$  (Ref. 11),  $\text{RuCl}_3$  (Ref. 12),  $\text{FeCl}_3$  (Ref. 13),  $\text{SmI}_2$  (Ref. 14),  $\text{Cp}_2\text{ZrH}_2/\text{NiCl}_2$  (Ref. 15),  $\text{KF}/\text{inorganic solid support}$ <sup>16</sup>, metal complexes<sup>17</sup> and molecular iodine<sup>18</sup>. Microwave irradiation methods

using  $\text{KF-Al}_2\text{O}_3$  (Ref. 19),  $\text{NaOH}$ <sup>20</sup>, acidic alumina<sup>21</sup> or  $\text{Na}_2\text{CO}_3$  (aq. solution)-TBAB (PTC condition)<sup>22</sup> as catalyst were also reported. However, the yields of the products in some of the aforesaid methods are not satisfactory. The procedures involve expensive reagents, tedious experimental procedures and longer durations. Molecular iodine, recently utilized<sup>18</sup> for rapid synthesis of  $\alpha, \alpha'$ -bis(arylmethylene)cycloalkanones, has hazardous effect<sup>23</sup> and it should be removed carefully from the reaction mixture by washing with aqueous  $\text{Na}_2\text{S}_2\text{O}_3$  solution. Thus, there is a need to develop a suitable and rapid method for synthesis of  $\alpha, \alpha'$ -bis(arylmethylene)cycloalkanones and related compounds using easily available, cheap and efficient catalyst. A survey of literature revealed that the reaction between cycloalkanones and aromatic aldehydes using amberlyst-15 as catalyst under microwave irradiation has not been studied so far. Herein, we describe an expeditious synthesis of  $\alpha, \alpha'$ -bis(arylmethylene)-cycloalkanones,  $\alpha, \alpha'$ -bis(cinnamylidene)cycloalkanones,  $\alpha$ -cinnamylideneacetophenones and chalcones applying amberlyst-15 under microwave irradiation. The catalyst, amberlyst-15 is easily available and the experimental procedure using this catalyst is very simple. As the catalyst works under heterogeneous condition it can easily be removed by filtration.

## Results and discussion

Various aromatic aldehydes were rapidly reacted with cyclohexanone and cyclopentanone when subjected to microwave irradiation in presence of neutral alumina supported amberlyst-15 under solvent free condition. The condensation proceeded smoothly to form  $\alpha, \alpha'$ -bis(arylmethylene)cyclohexanones (**3a-f**) and  $\alpha, \alpha'$ -bis(arylmethylene)cyclopentanones (**3g-k**), respectively, in very good yield (75–88%) (Scheme 1, Table 1). Furfuraldehyde and cinnamaldehyde also reacted with the said cyclic ketones in the similar way.

The experimental procedure is very simple and the conversion occurs in short irradiation time of *ca.* 3 min. After irradiation the mixture was cooled, shaken with small amount of chloroform and filtered. The filtrate was concentrated and subjected to rapid chromatography through silica gel column using petroleum ether-EtOAc (95 : 5) as eluent to afford the crystalline condensation product. The reactions were clean and free from side products such as self condensation of ketones. The presence of electron donating or electron withdrawing group in the aromatic aldehyde did not show any significant effect on



### Scheme 1

**Table 1.** Amberlyst-15 catalysed microwave assisted synthesis of  $\alpha,\alpha'$ -bis(arylmethylene)cycloalkanones and  $\alpha,\alpha'$ -bis(cinnamylidene)cycloalkanones



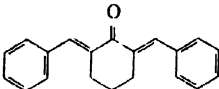

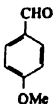
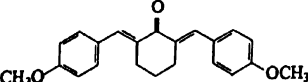


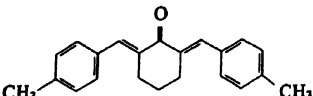

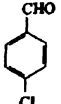
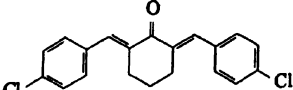


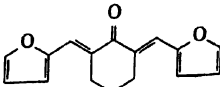


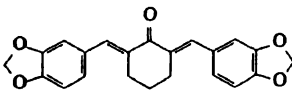


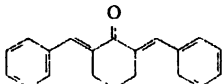


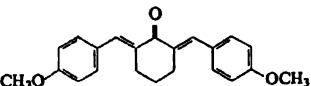

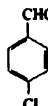
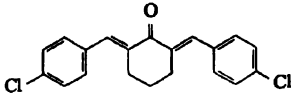


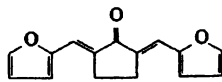


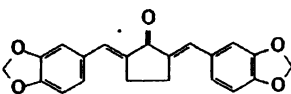

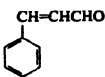
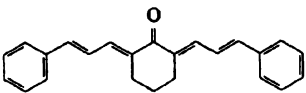

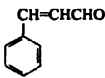
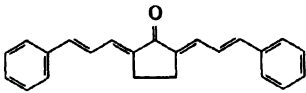
Entry	Ketone (1)	Aldehyde (2)	Product (3)	Isolated yield (%)	M.p. (°C)	Refs.
a				80	155	2
b				75	213	2
c				78	162–163	2
d				85	138	2
e				82	143–144	1b

Table-1 (contd.)

f				75	184	
g				86	190	2
h				75	215	2
i				85	229-230	2
j				79	204	18
k				78	262	-
l				80	178	19
m				85	220-221	19

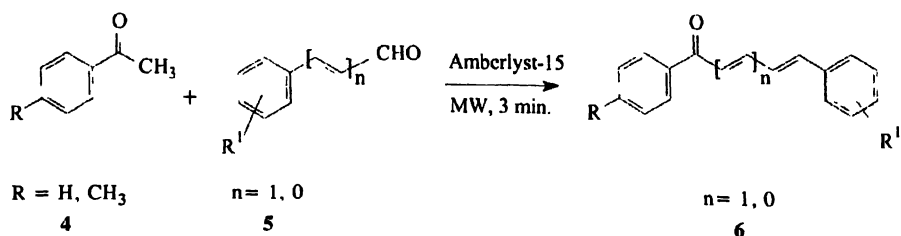
the rate of conversion under the present experimental condition. When this reaction was carried out under thermal condition in refluxing acetonitrile a longer time (10 h) was required and the yields of the products were also low (45–55%).

After getting success in condensation of cycloalkanones with aromatic aldehydes and related compounds, we turned our attention to apply this methodology for synthesis of chalcones and related compounds which are also well-known for their biological activities as well as for their easy transformation to other biologically active compounds<sup>1</sup>. Thus, when acetophenone and benzaldehyde or cinnamaldehyde were used, the desired chalcones and related compounds were obtained in 68–85% yield (Scheme 2, Table 2).

The catalytic role of amberlyst-15 in the above process was clearly evident from the observation that in its absence no reaction took place. Neutral alumina used acted as solid support only, and this view corroborates the findings of Esmaili *et al.*<sup>21</sup>.

#### Experimental

Melting points were recorded on a Kofler block and are uncorrected. IR spectra were recorded in KBr on a Perkin-Elmer FT-IR spectrometer (Spectrum RX 1). <sup>1</sup>H and <sup>13</sup>C NMR spectra were recorded in CDCl<sub>3</sub> on a Bruker AV-300 (300 MHz) spectrometer using TMS as internal standard. Analytical samples were routinely dried *in vacuo* at room temperature. Column chromatography was performed with silica gel (60–120 mesh) and TLC with silica



Scheme 2

Table 2. Amberlyst-15 catalysed microwave assisted synthesis of  $\alpha$ -cinnamylideneacetophenones and chalcones

Entry	Ketone (4)	Aldehyde (5)	Product (6)	Isolated yield (%)	M.p. (°C)	Refs.
a				68	86	-
b				70	120-121	-
c				69	76-78	-
d				73	136	-
e				75	78-80	-
f				68	56	24
g				72	68-70	25
h				75	110-112	25
i				85	112	-

gel G of SRL Pvt. Ltd. Neutral alumina used was also of SRL Pvt. Ltd. Petroleum ether had the boiling range 60–80 °C.

*General procedure for reaction of cycloalkanones and aldehydes under microwave irradiation :*

Cycloalkanones (1 mmol) and aldehydes (2 mmol) were mixed thoroughly with amberlyst-15 (100 mg) and neutral alumina (1 g) to form a powder. The mixture was taken in a glass conical flask (5 ml) and kept inside at the centre of a microwave oven (LG, DMO, Model No. MG-556P, 900 Watt) and the mixture was irradiated for *ca.* 3 min. For cyclopentanone 40% of the power level was applied but for other ketones 60% of the power level was applied to get maximum yield. After irradiation the mixture was cooled, shaken with chloroform (5 ml) and filtered. The filtrate was concentrated and subjected to rapid column chromatography over silica gel using petroleum ether-ethyl acetate (95 : 5) as eluent to obtain the crystalline pure product. Majority of the products were known compounds (referred in the Tables 1 and 2) and some of them were not known previously. The products were characterized from their literature melting points, physical, analytical and spectral (IR, <sup>1</sup>H NMR and <sup>13</sup>C) data. The spectral data of some of the compounds are given below :

**Compound 3f :** Yellow crystalline solid (yield : 75%), m.p. 184 °C; IR  $\nu_{\max}$  (KBr) : 1682 (C=O), 1595 (C=C)  $\text{cm}^{-1}$ ; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 300 MHz) :  $\delta$  1.76–1.84 (2H, quintet, *J* 6.3 Hz, H<sub>2</sub>-4 of cyclohexanone moiety), 2.87–2.92 (4H, dt, *J* 6.6 and 1.5 Hz, H<sub>2</sub>-3 and H<sub>2</sub>-5 of cyclohexanone moiety), 6.01 (4H, s, 2 × -OCH<sub>2</sub>O-), 6.85 (2H, d, *J* 8.4 Hz, 2 × H-5 of aryl moiety), 6.98 (2H, s, olefinic protons), 7.00 (2H, br. d, *J* 7.5 Hz, 2 × H-6 of aryl moiety), 7.70 (2H, br. s, 2 × H-2 of aryl moiety); <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) :  $\delta$  22.96, 28.51 (2C), 101.36 (2C), 108.43 (2C), 110.04 (2C), 125.80 (2C), 130.23 (2C), 134.69 (2C), 136.69 (2C), 147.72 (2C), 148.01 (2C), 190.05 (C=O) (Found : C, 72.64; H, 5.08. Calcd. for C<sub>22</sub>H<sub>18</sub>O<sub>5</sub> : C, 72.92; H, 5.01%).

**Compound 3k :** Yellow crystalline solid (yield : 78%), m.p. 262 °C; IR  $\nu_{\max}$  (KBr) : 1680 (C=O), 1600 (C=C)  $\text{cm}^{-1}$ ; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 300 MHz) :  $\delta$  3.04 (4H, s, H<sub>2</sub>-3 and H<sub>2</sub>-4 of cyclopentanone moiety), 6.05 (4H, s, 2 × -OCH<sub>2</sub>O-), 6.88 (2H, d, *J* 8.1 Hz, 2 × H-5 of aryl moi-

ety), 7.11 (2H, s, olefinic protons), 7.13 (2H, d, *J* 9.1 Hz, 2 × H-6 of aryl moiety), 7.50 (2H, br. s, 2 × H-2 of aryl moiety); <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) :  $\delta$  26.47 (2C), 101.52 (2C), 108.75 (2C), 109.75 (2C), 126.69 (2C), 130.35 (2C), 133.54 (2C), 135.54 (2C), 148.11 (2C), 148.72 (2C), 196.04 (C=O) (Found : C, 72.08, H, 4.75. Calcd. for C<sub>21</sub>H<sub>16</sub>O<sub>5</sub> : C, 72.41; H, 4.63%).

Condensation of acetophenones (1 mmol) with aldehydes (1 mmol) were done in the same way as described above. The products were characterized from their physical, analytical and spectral data. The spectral data of some of them are given below :

**Compound 6a :** Yellow crystalline solid (yield : 68%), m.p. 86 °C; IR  $\nu_{\max}$  (KBr) : 1660 (C=O), 1592 (C=C)  $\text{cm}^{-1}$ ; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 300 MHz) :  $\delta$  6.98–7.10 (2H, m, H-γ and H-δ), 7.09 (1H, d, *J* 15 Hz, H-α), 7.30–7.40 (3H, m, Ar-H), 7.47–7.52 (5H, m, Ar-H), 7.60 (1H, ddd, *J* 14.7, 7.5 and 5.1 Hz, H-β), 7.98 (2H, d, *J* 7.5 Hz, H-2 and H-5 of PhCO-) (Found : C, 86.72; H, 6.13. Calcd. for C<sub>17</sub>H<sub>14</sub>O : C, 87.15; H, 6.02%).

**Compound 6b :** Yellow crystalline solid (yield : 70%), m.p. 120–121 °C; IR  $\nu_{\max}$  (KBr) : 1655 (C=O), 1598 (C=C)  $\text{cm}^{-1}$ ; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 300 MHz) :  $\delta$  7.07–7.14 (3H, m, H-α, H-γ and H-δ), 7.36–7.43 (3H, m, H-3, H-4 and H-5 of C<sub>6</sub>H<sub>5</sub>-), 7.53 (2H, dd, *J* 7.8 and 1.5 Hz, H-2 and H-6 of C<sub>6</sub>H<sub>5</sub>-), 7.65–7.73 (2H, m, H-β and H-5 of *m*-C<sub>6</sub>H<sub>4</sub>NO<sub>2</sub>), 8.32 (1H, dd, *J* 7.8 and 1.2 Hz, H-4 or H-6 of *m*-O<sub>2</sub>N-C<sub>6</sub>H<sub>4</sub>-), 8.43 (1H, dd, *J* 8.1 and 1.2 Hz, H-6 or H-4 of *m*-O<sub>2</sub>N-C<sub>6</sub>H<sub>4</sub>-), 8.80 (1H, t, *J* 1.8 Hz, H-2 of *m*-O<sub>2</sub>N-C<sub>6</sub>H<sub>4</sub>-).

**Compound 6c :** Yellow crystalline solid (yield : 69%), m.p. 76–78 °C; IR  $\nu_{\max}$  (KBr) : 1651 (C=O), 1595 (C=C)  $\text{cm}^{-1}$ ; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 300 MHz) :  $\delta$  3.90 (3H, s, OCH<sub>3</sub>), 6.98 (2H, d, *J* 8.8 Hz, H-3 and H-5 of *p*-MeOC<sub>6</sub>H<sub>4</sub>-), 6.99–7.04 (2H, m, H-γ and H-δ), 7.12 (1H, d, *J* 15.1 Hz, H-α), 7.30–7.41 (3H, m, H-3, H-4 and H-5 of C<sub>6</sub>H<sub>5</sub>-), 7.51 (2H, dd, *J* 8.1 and 1.5 Hz, H-2 and H-6 of C<sub>6</sub>H<sub>5</sub>-), 7.60 (1H, ddd, *J* 15.3, 7.2 and 3.0 Hz, H-β), 8.00 (2H, d, *J* 8.6 Hz, H-2 and H-6 of *p*-MeOC<sub>6</sub>H<sub>4</sub>-); <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) :  $\delta$  55.42 (OCH<sub>3</sub>), 113.76 (2C), 125.25, 127.02, 127.17 (2C), 128.77 (2C), 129.03, 130.62 (2C), 131.01, 136.17, 141.33, 143.93, 163.29, 188.66 (C=O).

**Compound 6d** : Yellow crystalline solid (yield : 73%), m.p. 136 °C; IR  $\nu_{\max}$  (KBr) : 1663 (C=O), 1598 (C=C)  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 300 MHz) :  $\delta$  7.02–7.07 (3H, m, H- $\alpha$ , H- $\gamma$  and H- $\delta$ ), 7.30–7.41 (3H, m, H-3, H-4 and H-5 of  $\text{C}_6\text{H}_5$ -), 7.46 (2H, d,  $J$  8.4 Hz, H-3 and H-5 of  $p\text{-Cl-C}_6\text{H}_4$ -), 7.51 (2H, dd,  $J$  8.1 and 1.5 Hz, H-2 and H-6 of  $\text{C}_6\text{H}_5$ -), 7.60 (1H, ddd,  $J$  14.7, 7.0 and 3.2 Hz, H- $\beta$ ), 7.92 (2H, d,  $J$  8.4 Hz, H-2 and H-6 of  $p\text{-Cl-C}_6\text{H}_4$ -).

**Compound 6e** : Yellow crystalline solid (yield : 75%), m.p. 78–80 °C; IR  $\nu_{\max}$  (KBr) : 1652 (C=O), 1600 (C=C)  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 300 MHz) :  $\delta$  2.43 (3H, s,  $\text{CH}_3$ ), 6.97–7.05 (2H, m, H- $\gamma$  and H- $\delta$ ), 7.10 (1H, d,  $J$  17.2 Hz), 7.27–7.40 (5H, m, H-3, H-4 and H-5 of  $\text{C}_6\text{H}_5$ -, and H-3 and H-5 of  $p\text{-Me-C}_6\text{H}_4$ -), 7.50 (2H, dd,  $J$  6.9 and 1.3 Hz, H-2 and H-6 of  $\text{C}_6\text{H}_5$ -), 7.60 (1H, ddd,  $J$  15.3, 7.2 and 3.0 Hz, H- $\beta$ ), 7.89 (2H, d,  $J$  8.1 Hz, H-2 and H-6 of  $p\text{-Me-C}_6\text{H}_4$ -).

**Compound 6i** : Light yellow crystalline solid (yield : 85%), m.p. 112 °C; IR  $\nu_{\max}$  (KBr) : 1675 (C=O), 1590 (C=C)  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 300 MHz) :  $\delta$  2.43 (3H, s,  $\text{CH}_3$ ), 6.00 (2H, s,  $-\text{OCH}_2\text{O}-$ ), 6.83 (1H, d,  $J$  7.8 Hz, H-5), 7.11 (1H, dd,  $J$  8.0 and 1.5 Hz, H-6), 7.17 (1H, d,  $J$  1.5 Hz), 7.29 (2H, d,  $J$  8.1 Hz, H-3' and H-5'), 7.37 (1H, d,  $J$  15.6 Hz, H- $\alpha$ ), 7.73 (1H, d,  $J$  15.6 Hz, H- $\beta$ ), 7.92 (2H, d,  $J$  8.1 Hz, H-2' and H-6');  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ ) :  $\delta$  21.65 ( $\text{CH}_3$ ), 101.60, 106.66, 108.65, 120.15, 125.09, 128.56 (2C), 129.29 (2C), 129.48, 135.81, 143.46, 144.23, 148.39, 149.81, 189.89 (C=O) (Found : C, 76.55; H, 5.19. Calcd. for  $\text{C}_{17}\text{H}_{14}\text{O}_3$  : C, 76.68; H, 5.30%).

#### Conclusion :

A very convenient and facile method for synthesis of  $\alpha, \alpha'$ -bis(arylmethylene)cycloalkanones,  $\alpha, \alpha'$ -bis(cinnamylidene)cycloalkanones,  $\alpha$ -cinnamylideneacetophenones and chalcones by the reaction of appropriate ketones and aldehydes using amberlyst-15 under microwave irradiation has been developed. The rapid conversion of starting materials, very good yields of products, utilization of an inexpensive catalyst and operational simplicity are the great advantages of the present procedure. The combination of the beneficial effect derived from the heterogeneous catalyst and microwave irradiation has properly been utilized here to make the process highly efficient. The procedure is environmentally benign.

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