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# MICROWAVE-ASSISTED CLAISEN-SCHMIDT CONDENSATION BETWEEN ARYLMETHYL KETONES AND ARYL ALDEHYDES CATALYZED BY CU(OTF)$_2$ UNDER SOLVENT-FREE CONDITIONS: SYNTHESIS OF CHALCONES

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**Abstract.** An environmentally friendly and high yield method for the Claisen-Schmidt condensation has been investigated. A mixture of an arylmethyl ketone and an aldehyde was irradiated in a microwave reactor for 20 minute using Cu(OTf)$_2$ as catalyst in solvent-free conditions. 12 chalcones were synthesized in moderate to excellent yields (74 - 91 %). This is the first time the Claisen-Schmidt condensation with Cu(OTf)$_2$ under microwave irradiation is reported.

*Keywords:* bioactivity, benzaldehyde, acetophenone, irradiation, copper triflate.

*Classification numbers:* 1.1.3, 1.1.6, 2.6.1.

## 1. INTRODUCTION

The aldol condensation reaction is one of the most fundamental carbon-carbon bond-forming reactions in organic chemistry and widely used in the production of various chemicals including pharmaceutical compounds [1 - 3]. The reaction produces β-hydroxy aldehydes or β-hydroxy ketones by self-condensation or cross condensation of carbonyl compounds as well as provides α,β- unsaturated aldehydes or α,β-unsaturated ketones formed from dehydrating of these β-hydroxy carbonyl compounds. If one of two reactants is an aromatic carbonyl compound lacking an alpha-hydrogen, the reaction is called Claisen-Schmidt condensation. This reaction is named after two of its pioneering investigators R. L. Claisen and J. G. Schmidt, who independently published on this topic in 1880 and 1881 [4, 5]. Among Claisen-Schmidt reaction products, chalcones, whose general structure is outlined in Figure 1, are considered the most important compounds. They demonstrate numerous biological activities such as anti-diabetic, anti-neoplastic, anti-hypertensive, anti-retroviral, anti-inflammatory, anti-parasitic, anti-histaminic, anti-malarial, anti-oxidant, anti-fungal, anti-obesity, anti-platelet, anti-tubercular, immunosuppressant, anti-arrhythmic, hypnotic, anti-gout, anxiolytic, anti-spasmodic, anti-nociceptive, hypolipidemic, anti-filarial, anti-angiogenic, anti-protozoal, anti-bacterial, anti-steroidal, and cardioprotective [6 - 9].

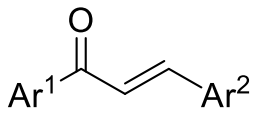



*Figure 1*. General structure of chalcones.

Many types of catalyst such as acid catalysts, base catalysts, organocatalysts, metal catalysts, and biocatalysts have been employed for the Aldol condensation [2 - 3, 10 - 14]. Lewis acids including copper triflate ($Cu(OTf)_2$) also have been commonly used as catalysts for this reaction [15 - 16].

The recent development in so called ‘‘green chemistry’’ shows that alternative methods of carrying out chemical harmfulness of classical reactions. Besides using less catalyst and less or without solvent, one of the most popular and interesting approaches in this field is employing the microwave energy for conducting many chemical transformations. The interaction of the matter with such kinds of electromagnetic waves results in higher speed of heating [17], much shorter reaction time and very often the higher selectivity of desired products. Recently, some studies about aldol condensation under microwave conditions have also been reported but most of them were carried in organic solvent. Herein, we investigated a microwave-assisted direct Claisen-Schmidt condensation catalyzed by $Cu(OTf)_2$ under solvent free conditions between arylmethyl ketones and aryl aldehydes to synthesize chalcones. Previously, we already used $Cu(OTf)_2$ as a catalyst for the Baeyer-Villiger oxidation reaction to convert ketone to lactones or ester [18].

## 2. MATERIALS AND METHODS

### 2.1. Experimental section

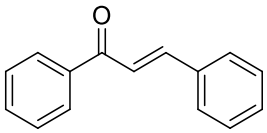
#### *2.1.1. General procedure*

All chemicals were purchased from Sigma- Aldrich company. Microwave reactions were performed in a CEM microwave reactor at 120 °C, 150 W in a 3 mL capped vial. Column chromatography was performed using Merck silica gel (40 - 63 μm) packed by the slurry method, under a positive pressure of air. $^1H$ and $^{13}C$ NMR spectra were recorded on a Varian Inova NMR Spectrometer ($^1H$ NMR running at 400 MHz and $^{13}C$ NMR running at 100 MHz) instrument. $CDCl_3$ was used as the NMR solvent. Data for $^1H$ NMR are reported as follows: chemical shift (δ ppm), integration, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), coupling constant and assignment. Data for $^{13}C$ NMR are reported in terms of chemical shift (δ ppm).

General procedure for Claisen-Schmidt condensation: Arylmethyl ketone (2 mmol) was placed in a microwave vial. The corresponding Aryl aldehyde (2.4 mmol, 1.2 equiv) and $Cu(OTf)_2$ (12 mg, 0.04 mmol, 0.02 equiv) were added consecutively. The reaction mixture was left stirring under microwave irradiation (initial setting at 150 W) for 20 minutes at 120 °C. The reaction mixture then was dissolved in diethyl ether (50 ml) and washed with brine (2 × 20 ml). The organic layer was dried over $MgSO_4$ and filtered. Solvent then was evaporated from the organic layer and the residue was purified using column chromatography (Eluent: n-hexane/$Et_2O$) to give the desired product. The yields of chalcone products were calculated according to arylmethyl ketones ((mole of isolated chalcone/ mole of arylmethyl used) × 100).

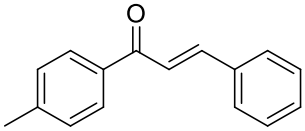
#### *2.1.2. NMR data for synthesized compounds*

**(*E*)-1,3-Diphenyl-2-propen-1-one (product 3)**



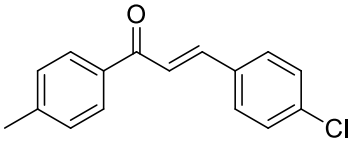
362 mg, 87 %, yellowish solid, $^{1}$H NMR (400 MHz, $CDCl_3$) δ 8.01 - 8.05 (m, 2H), 7.82 (d, *J* = 15.6 Hz,1H), 7.63 - 7.68 (m, 2H), 7.57 - 7.62 (m, 1H), 7.49 - 7.57 (m, 3H), 7.41 - 7.45 (m, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 190.7, 144.8, 138.4, 135.0, 132.9, 130.7, 129.1, 128.8, 128.6, 128.5, 122.3. NMR data are consistent with literature report [16].

**(*E*)-3-Phenyl-1-(*p*-tolyl)prop-2-en-1-one (table 3, entry 1, product 4)**



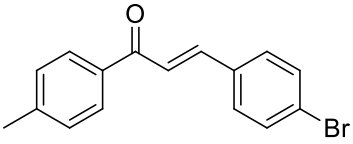
382 mg, 86 %, yellowish solid, $^{1}$H NMR (400 MHz, $CDCl_3$) δ 7.95 (d, *J* = 7.6 Hz, 2H), 7.81 (d, *J* = 15.6 Hz, 1H), 7.67–7.63 (m, 2H), 7.55 (d, *J* = 15.6 Hz, 1H), 7.45 - 7.40 (m, 3H), 7.31 (d, *J* = 7.6 Hz, 2H), 2.44 (s, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 190.1, 144.5, 143.8, 135.7, 135.1, 130.5, 129.4, 129.0, 128.8, 128.5, 122.2, 21.8, NMR data are consistent with literature report [19].

**(*E*)-3-(4-Chlorophenyl)-1-(*p*-tolyl)prop-2-en-1-one (table 3, entry 2, product 5)**



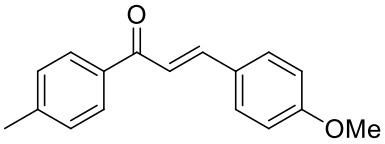
385 mg, 75 %, yellowish solid, $^{1}$H NMR (400 MHz, $CDCl_3$) δ 7.93 (d, *J* = 8.4 Hz, 2H), 7.75 (d, *J* = 15.6 Hz, 1H), 7.57 (d, *J* = 8.4 Hz, 2H), 7.50 (d, *J* = 15.6 Hz, 1H), 7.39 (d, *J* = 8.4 Hz, 2H), 7.31 (d, *J* = 8.4 Hz, 2H), 2.44 (s, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 189.9, 144.0, 143.0, 136.4, 135.6, 133.7, 129.7, 129.5, 129.4, 128.8, 122.7, 21.8. NMR data are consistent with literature report [19].

**(*E*)-3-(4-Bromophenyl)-1-(*p*-tolyl)prop-2-en-1-one (table 3, entry 3, product 6)**



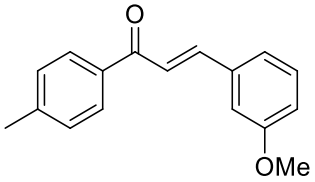
463 mg, 77 %, yellowish solid, $^{1}$H NMR (400 MHz, $CDCl_3$) δ 7.93 (d, *J* = 8.0 Hz, 2H), 7.73 (d, *J* = 15.6 Hz, 1H), 7.57-7.48 (m, 5H), 7.30 (d, *J* = 8.0 Hz, 2H), 2.44 (s, 3H) ; $^{13}$C NMR (100 MHz, $CDCl_3$) δ 189.8, 144.0, 143.0, 135.6, 134.1, 132.3, 129.9, 129.5, 128.8, 124.8, 122.8, 21.8. NMR data are consistent with literature report [19].

**(*E*)-3-(4-Methoxyphenyl)-1-(*p*-tolyl)prop-2-en-1-one (table 3, entry 4, product 7)**



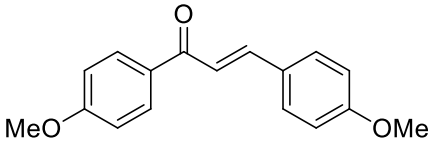

423 mg, 84 %, yellowish solid, $^{1}$H NMR (400 MHz, $CDCl_3$) δ 7.93 (d, *J* = 8.4 Hz, 2H), 7.78 (d, *J* = 15.6 Hz, 1H), 7.60 (d, *J* = 8.4 Hz, 2H), 7.42 (d, *J* = 15.6 Hz, 1H), 7.29 (d, *J* = 8.4 Hz, 2H), 6.93 (d, *J* = 8.4 Hz, 2H), 3.85 (s, 3H), 2.43 (s, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 190.2, 161.7, 144.4, 143.5, 136.1, 132.1, 129.4, 128.7, 127.9, 120.0, 114.5, 55.5, 21.8. NMR data are consistent with literature report [19].

**(*E*)-3-(3-Methoxyphenyl)-1-(*p*-tolyl)prop-2-en-1-one (table 3, entry 5, product 8)**



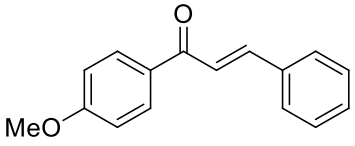

418 mg, 83 %, yellowish solid, $^{1}$H NMR (400 MHz, $CDCl_3$) δ 7.93 (d, *J* = 8 Hz, 2H), 7.76 (d, *J* = 15.6 Hz, 1H), 7.51 (d, *J* = 15.6 Hz, 1H), 7.31-7.36 (m, 2H), 7.30 (s, 1H), 7.24 (d, *J* = 7.6 Hz, 1H), 7.16 (t, *J* = 2.4 Hz, 1H), 6.98 - 6.94 (m, 1H), 3.86 (s, 3H), 2.44 (s, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 190.2, 160.1, 144.5, 143.8, 135.8, 136.6, 130.1, 129.5, 128.8, 122.6, 121.2, 116.3, 113.6, 55.5, 21.8. NMR data are consistent with literature report [19].

**(*E*)-1,3-Bis(4-methoxyphenyl)prop-2-en-1-one (table 3, entry 6, product 9)**




434 mg, 81 %, yellowish solid, $^{1}$H NMR (400 MHz, $CDCl_3$) δ 8.03 (d, *J* = 8.8 Hz, 2H), 7.78 (d, *J* = 15.6 Hz, 1H), 7.60, (d, *J* = 8.8 Hz, 2H), 7.43 (d, *J* = 15.6 Hz, 1H), 6.98 (d, *J* = 8.8 Hz, 2H), 6.93 (d, *J* = 8.8 Hz, 2H), 3.88 (s, 3H), 3.85 (s, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 189.0, 163.4, 161.7, 144.0, 131.5, 130.8, 130.2, 128.0, 119.8, 114.5, 113.9, 55.6, 55.5. NMR data are consistent with literature report [17].

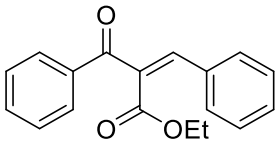
**(*E*)-1-(4-Methoxyphenyl)-3-phenylprop-2-en-1-one (table 3, entry 7, product 10)**




405 mg, 85 %, yellowish solid, $^{1}$H NMR (400 MHz, $CDCl_3$) δ 8.05 (d, *J* = 8.8 Hz, 2H), 7.81 (d, *J* = 15.6 Hz, 1H), 7.67–7.63 (m, 2H), 7.55 (d, *J* = 15.6 Hz, 1H), 7.39–7.45 (m, 3H), 6.99 (d, *J*=

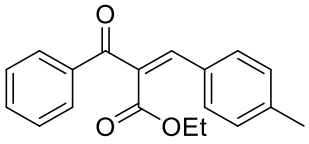
8.8 Hz, 2H), 3.89 (s, 3H), ; $^{13}$C NMR (100 MHz, $CDCl_3$) δ 188.9, 163.6, 144.1, 135.2, 131.3, 131.0, 130.5, 129.1, 128.5, 122.1, 114.0, 55.6. NMR data are consistent with literature report [16].

**Ethyl (E)-2-benzoyl-3-phenylacrylate (table 3, entry 8, product 11)**



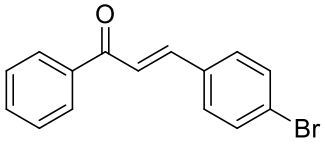

510 mg, 91 %, yellowish solid, $^1$H NMR (400 MHz, CDCl3) δ 7.97–7.92 (m, 3H), 7.59–7.53 (m, 1H), 7.46 - 7.40 (m, 2H), 7.38–7.33 (m, 2H), 7.27–7.20 (m, 3H), 4.21 (q, *J* = 7.2 Hz, 2H), 1.16 (t, *J* = 7.2 Hz, 3H), ; $^{13}$C NMR (100 MHz, $CDCl_3$) δ 195.8, 165.2, 142.7, 136.3, 134.0, 133.0, 131.5, 130.5, 130.3, 129.3, 129.0, 128.9, 61.7, 14.2. NMR data are consistent with literature report [20].

**Ethyl (E)-2-benzoyl-3-phenylacrylate (table 3, entry 9, product 12)**



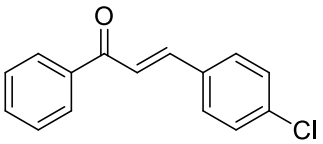

523 mg, 89 %, yellowish liquid, $^1$H NMR (400 MHz, $CDCl_3$) δ 7.91–7.86 (m, 2H), 7.85 (s, 1H), 7.51–7.47 (m, 1H), 7.36 (t, *J* = 7.6 Hz, 2H), 7.21–7.17 (m, 3H), 6.99–6.93 (m, 2H), 4.14 (q, *J* = 7.2 Hz, 2H), 1.09 (t, *J* = 7.2 Hz, 3H), ; $^{13}$C NMR (100 MHz, $CDCl_3$) δ 196.0, 165.2, 142.9, 141.2, 136.5, 133.9, 130.6, 130.4, 130.2, 129.8, 129.4, 128.9, 61.7, 21.6, 14.2. NMR data are consistent with literature report [20].

**(*E*)-3-(4-Chlorophenyl)-1-phenylprop-2-en-1-one (table 3, entry 10, product 13)**




431 mg, 75 %, yellowish solid, $^1$H NMR (400 MHz, $CDCl_3$) *δ* 8.03 - 8.02 (m, 2H), 7.49 (d, *J* = 15.6 Hz, 1H), 7.61 - 7.55 (m, 3H), 7.52 - 7.48 (m, 3H), 7.40 - 7.37 (m, 2H); $^{13}$C NMR (100 MHz, $CDCl_3$) *δ* 190.1, 143.2, 138.0, 136.4, 133.3, 132.9, 129.5, 129.2, 128.6, 128.4, 122.4.NMR data are consistent with literature report [21].

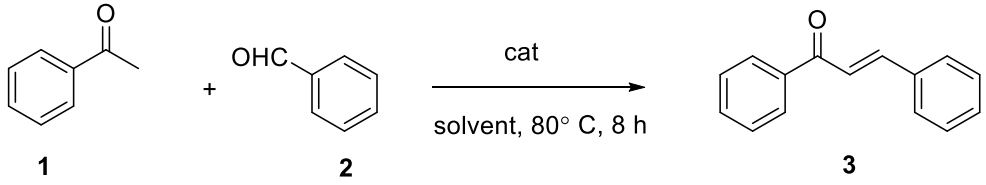
**(*E*)-3-(4-bromophenyl)-1-phenylprop-2-en-1-one (Table 3, entry 11, product 14)**



359 mg, 74 %, white solid, $^1$H NMR (400 MHz, $CDCl_3$) δ 8.04 (d, *J* = 8.0 Hz, 2H), 7.79 (d, *J* = 15.6 Hz, 1H), 7.63-7.60 (m, 3H), 7.56 - 7.52 (m, 3H), 7.42 (d, *J* = 8.0 Hz, 2H). $^{13}$C NMR (100 MHz, $CDCl_3$) *δ* 190.3, 143.3, 138.1, 136.5, 133.4, 132.9, 129.6, 129.3, 128.7, 128.5, 122.5. NMR data are consistent with literature report [21].

## 3. RESULTS AND DISCUSSION

Initially, we tested effects of some metal triflate catalysts and solvents to the Claisen-Schmidt condensation with conventional heating. As the outset of this study, we employed acetophenone **1** (2 mmol) and benzaldehyde **2** (2.4 mmol, 1.2 equiv) as the model substrate in different solvents or without solvent to optimize the reaction conditions. Reaction mixtures were heated at 80 °C for 8 h. The results are shown in the Table 1.




*Scheme 1*. Claisen-Schmidt condensation with different metal triflate catalysts.

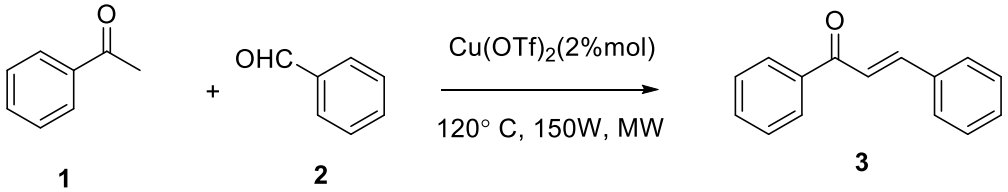
*Table 1*. Claisen-Schmidt condensation between acetophenone and benzaldehyde with metal triflate catalysts.

| Entry | Solvent | Catalyst | Amount of | Yield (%) |
|---|---|---|---|---|
| 1 | $CH_3CN$ | $Zn(OTf)_2$ | 5 | 46 |
| 2 | $CH_3CN$ | $Sc(OTf)_3$ | 5 | 52 |
| 3 | $CH_3CN$ | $Cu(OTf)_2$ | 5 | 83 |
| 4 | $C_2H_5OH$ | $Zn(OTf)_2$ | 5 | 24 |
| 5 | $C_2H_5OH$ | $Sc(OTf)_2$ | 5 | 27 |
| 6 | $C_2H_5OH$ | $Cu(OTf)_2$ | 5 | 65 |
| 7 | THF | $Cu(OTf)_2$ | 5 | 51 |
| 8 | Toluene | $Cu(OTf)_2$ | 5 | 57 |
| 9 | $CH_3CN$ | $Cu(OTf)_2$ | 2 | 82 |
| 10 | None | $Cu(OTf)_2$ | 2 | 84 |

Among three metal triflates, $Cu(OTf)_2$ showed the best catalytic effects in both solvents, $CH_2CN$ and $C_2H_5OH$ (entry 1-6). Then we tried the reaction with two other different solvents (entry 7 - 8) using $Cu(OTf)_2$ and $CH_3CN$ was proved to be the most suitable solvent for this transformation (entry 3). Reducing the amount of catalyst $Cu(OTf)_2$ from 5 % to 2 % did not influence much to the yield of the reaction (entry 9). Noticeably, reaction yield was even slightly improved in solventless conditions (entry 10).

Concerning more about environmental effects, we then investigated the Claisen-Schmidt

condensation catalyzed by $Cu(OTf)_2$ with microwave irradiation. Mixture of acetophenone (2 mmol) and benzaldehyde (2.4 mmol, 1.2 equiv) was irradiated at 120 °C, 150 W in solventless conditions for stated time and the results were summarized in the Table 2.



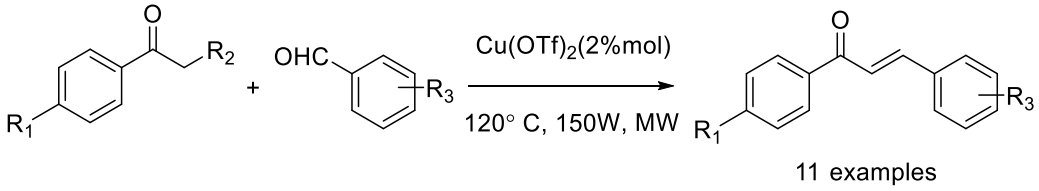

*Scheme 2*. Reaction optimization under microwave irradiation.

*Table 2*. Microwave-assisted aldol condensation between acetophenone and benzaldehyde.

| Entry | Solvent | Time | Yield (%) |
|---|---|---|---|
| 1 | none | 30 min | 87 |
| 2 | none | 20 min | 87 |
| 3 | none | 10 min | 82 |

In solvent-free conditions, the product was formed in 87 % yield after 30 minutes of irradiation (Table 2, entry 1). The same yield was observed when irradiation time was reduced to 20 min (Table 2, entry 2). Within 10 min of microwave irradiation, reaction yield decreased to 8 2 % (Table 2, entry 3). We concluded that 20 min of irradiation is sufficient for the reaction. So solvent-free, 0.02 equivalent of catalyst, and 20 minutes of irradiation at 120 °C and 150 W were the optimal conditions of our design for the reaction.

With environmentally friendly design in hand, we expanded the reaction for other arylmethyl ketones and aryl aldehydes. The results were given in Table 3. Reaction yields range from 74 % to 91 % for various substrates. Introduction of COOEt to α position of aryl ketones improved the yields slightly because of the ease of formation of the corresponding enolate anion (entry 8, 9). Electron-donating groups in benzene ring of both reactants led to a slight decrease in reaction yields (entry 1, 4, 5, 6, 7). Surprisingly, lower yields were observed with the presence of electron-withdrawing groups (Br, Cl) in the aldehyde moiety (entry 2, 3, 10, 11). In general, strong electron-withdrawing groups such as F, $NO_2$ in the benzene ring of both aldehydes and ketones will accelerate the Claisen-Schmidt condensation. In contrast, electron-donating groups in the benzene ring of both aldehydes and ketones will slightly deactivate the reaction. In our study, most reaction yields are consistent with this rule. However, we are not sure why reaction yields in entries 2, 3, 10, and 11 are not high.




*Scheme 3*. Synthesis of various chalcones with microwave irradiation.

*Table 3*. Synthesis of chalcones under microwave irradiation with $Cu(OTf)_2$ catalyst.

| Entry | $R_1$, $R_2$ | $R_3$ | Product | Yield |
|---|---|---|---|---|
| 1 | Me, H | H | 4 | 86 |
| 2 | Me, H | 4-Cl | 5 | 75 |
| 3 | Me, H | 4-Br | 6 | 77 |
| 4 | Me, H | 4-OMe | 7 | 84 |
| 5 | Me, H | 3-OMe | 8 | 83 |
| 6 | OMe, H | 4-OMe | 9 | 81 |
| 7 | OMe, H | H | 10 | 85 |
| 8 | H, COOEt | H | 11 | 91 |
| 9 | H, COOEt | 4-Me | 12 | 89 |
| 10 | H, H | 4-Br | 13 | 75 |
| 11 | H, H | 4-Cl | 14 | 74 |

## 4. CONCLUSIONS

We have investigated the Claisen- Schmidt condensation between arylmethyl ketones and aryl aldehydes catalyzed by copper triflate in different conditions and designed the optimal conditions. The optimal conditions were environmentally friendly: solvent free, short time of heating and high yields of products. 12 chalcones were synthesized in moderate to excellent yield (74 - 91 %) following the optimal condition. Because of time constraint, only aryl aldehydes were studied. In future, the reaction will be expanded for aliphatic aldehydes. Reaction mechanism is in progress in our lab and will be reported in due course.

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***CRediT authorship contribution statement.*** Dau Xuan Duc: Methodology, Investigation, Data collecting and analysis. Nguyen Thi Chung: Introduction, Formal analysis, References.

***Declaration of competing interest.*** The authors declare that we have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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