

METHODOLOGY

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Microwave assisted solvent free synthesis of 1,3-diphenylpropenones

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Abstract

Background: 1,3-Diphenylpropenones (chalcones) are well known for their diverse array of bioactivities. Hydroxyl group substituted chalcones are the main precursor in the synthesis of flavonoids. Till date various methods have been developed for the synthesis of these very interesting molecules. Continuing our efforts for the development of simple, eco-friendly and cost-effective methodologies, we report here a solvent free condensation of aryl ketones and aldehydes using iodine impregnated alumina under microwave activation. This new protocol has been applied to a variety of substituted aryl carbonyls with excellent yield of substituted 1,3-diphenylpropenones.

Results: Differently substituted chalcones were synthesized using iodine impregnated neutral alumina as catalyst in 79-95% yield in less than 2 minutes time under microwave activation without using any solvent. The reaction was studied under different catalytic conditions and it was found that molecular iodine supported over neutral alumina gives the best yield. The otherwise difficult single step condensation of hydroxy substituted aryl carbonyls is an attractive feature of this protocol to obtain polyhydroxychalcones in excellent yields. In order to find out the general applicability of this new endeavor it was successfully applied for the synthesis of 15 different chalcones including highly bioactive prenylated hydroxychalcone xanthohumol.

Conclusion: A new, simple and solvent free method was developed for the synthesis of substituted chalcones in environmentally benign way. The mild reaction conditions, easy work-up, clean reaction profiles render this approach as an interesting alternative to the existing methods.

Background

1,3-Diphenylpropenones (chalcones) exhibit a broad spectrum of biological activities [1]. These are the main precursor in the biosynthesis of flavonoids [2] abundant in edible plants. They have been reported to show various pharmacological activities like anticancer [3,4], anti-malarial [5], anti-inflammatory [6], anti-tubercular [7], cytotoxic [8], gastroprotective [9], modulation of nitric oxide production [10] and so on. These compounds are important synthons for the preparation of five and six membered ring systems [11] as well as intermediate in the synthesis of many pharmaceuticals [12]. Having such a varied pharmacological activity and synthetic utility, chalcones have attracted chemists to develop newer strategies for their synthesis.

By far the most popular way of synthesis of chalcone is the Claisen-Schmidt condensation of an appropriate

acetophenone with benzaldehyde in presence of aqueous bases like NaOH [13-15], KOH [16], Ba(OH)₂ [17,18] etc. Other base catalysts such as magnesium t-butoxide [19], potassium carbonate [20], alumina [21], MgO [22], calcinated hydrotalcites [23,24], natural phosphate/NaNO₃ [25,26], KF/natural phosphate [27] and piperidine [28] have also been used for their synthesis.

The various activities of chalcones are largely dependent on the number and positions of hydroxy, methoxy and other substituent groups in both A and B rings [29]. Hydroxy chalcones are the main synthon for the synthesis of a number of naturally occurring bioactive flavonoids [1]. Literature data reveals that presence of hydroxyl substituent on the benzaldehyde aromatic ring hinders the base catalyzed aldol reaction. This happens because of the decreased reactivity of the carbonyl component due to the delocalization of the phenoxide anion formed [30]. Thus it becomes necessary to use protecting groups to stop the formation of the phenoxide ion in the preparation of hydroxychalcones under basic

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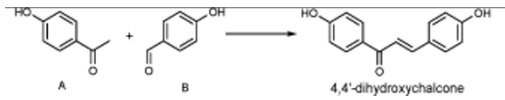
conditions [31,32]. This problem can be overcome by using acid catalysts like HCl, BF₃, B₂O₃, PTSA, SOCl₂/EtOH [30], AlCl₃ [33], BF₃-Et₂O [34], TiCl₄ [35], zeolites [36], RuCl₃ [37], Bronsted acidic ionic liquids [38] and H₂SO₄ in AcOH [39], but many of them suffer from the drawbacks of lower yields and harsh, environmentally detrimental reaction conditions.

In an ongoing project on the synthesis of bio-active molecules, we required a number of hydroxychalcones. The results obtained using existing methods were not satisfactory. We therefore tried some new catalysts for this conversion including iodine-alumina because of our previous experience of using iodine as an inexpensive, nontoxic, readily available catalyst in many other reactions [40-44]. Due to stringent and growing environmental regulations, the chemical industry needs the development of more eco-compatible synthetic methodologies [45]. The use of heterogeneous catalysts under solvent free conditions represents a potentially valuable and clean route to a range of organic products [46]. Microwave assisted synthetic reactions are gaining importance in recent years because of its endorsement under Green chemistry protocol [47,48]. We found iodine-alumina to be an excellent catalyst for the synthesis of chalcones under microwave irradiation giving 79-95% yield in a very short reaction time. During the preparation of this manuscript, Sashidhara et al [49] reported the synthesis of chalcones using iodine as catalyst but their method suffers from the requirement of dry dioxane as solvent and longer reaction time.

Results and discussions

As we were emphasizing solvent less single step synthesis of hydroxy chalcones without using any protecting group, the reaction between 4'-hydroxyacetophenone (A) and 4-hydroxybenzaldehyde (B) was studied in detail to optimize the reaction conditions. In order to find out the most effective condensation, equimolar mixtures of A and B was exposed to microwave irradiation in different reaction conditions. We studied the reaction using neutral alumina, acidic alumina, basic alumina, molecular iodine, iodine-neutral alumina, iodine-acidic alumina, iodine-basic alumina and without any catalyst. The results are presented in table 1, from which it can be found that molecular iodine impregnated neutral alumina gives the best yield (94%) after only 80 seconds microwave irradiation at 60°C, 120 W. The use of microwave irradiation was found obligatory as a control reaction carried out at 60°C keeping the other reaction conditions unchanged, yields only traces of the product (entry 10, table 1). It is believed that, I₂ being a Lewis acid, facilitates the enolisation of the aryl ketone as well as activates the carbonyl carbon of the benzaldehyde

Table 1 Condensation of A and B under different reaction conditions



Entry	Catalyst	Catalyst loading ^a	Time (sec)	Yield ^b (%)
1	None	-	100	Trace
2	Al ₂ O ₃ (neutral)	1:1	100	9
3	Al ₂ O ₃ (basic)	1:1	100	23
4	Al ₂ O ₃ (acidic)	1:1	100	27
5	I ₂	10	100	63
6	I ₂	20	100	65
7	I ₂	50	100	61
8	I ₂ -Al ₂ O ₃ (basic)	1:1	100	67
9	I ₂ -Al ₂ O ₃ (neutral)	1:1	100	94
10	I ₂ -Al ₂ O ₃ (neutral)	1:1	300	Trace ^c
11	I ₂ -Al ₂ O ₃ (acidic)	1:1	100	71
12	I ₂ -Al ₂ O ₃ (neutral)	2:1	100	69
13	I ₂ -Al ₂ O ₃ (neutral)	2:1	150	69
14	I ₂ -Al ₂ O ₃ (neutral)	2:1	300	71
15	I ₂ -Al ₂ O ₃ (neutral)	1:2	80	94
16	I ₂ -Al ₂ O ₃ (neutral)	1:2	60	81

^a For entry 5-7, catalyst loading is in mol%. For others it is given as substrate (A): catalyst (w/w).

^b Isolated pure yield; unreacted starting materials recovered.

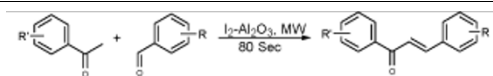
^c The reaction mixture was kept in a preheated oil bath at 60°C for the specified time.

towards nucleophilic attack. Upon impregnation over alumina, the effective surface area of the catalyst increases for the reaction to occur.

After selecting the catalyst, we studied the effect of different catalyst loadings on the yield and time of the reaction. The best result was obtained using 200 mg of iodine-neutral alumina for 100 mg of the ketone i.e. 1:2 (w/w), substrate:catalyst (Entry 15, Table 1), where the amount of I₂ is 10 mg (0.04 mmol) and Al₂O₃ is 190 mg (1.86 mmol). We further studied another reaction using same amount of I₂ and Al₂O₃ without impregnation keeping other reaction conditions unchanged but only 11% product formation was observed. It signifies the importance of impregnation of iodine with alumina showing a synergistic effect between them in catalyzing the reaction. In this optimized reaction conditions we proceeded to evaluate the general applicability of this method with a variety of ketones and aldehydes as shown in Table 2.

The electron donating and electron withdrawing substituents in the aryl ring of the ketones or of the aldehydes were well tolerated to give moderate to high yields of the desired chalcones. In general the reaction was clean and no side products were detected.

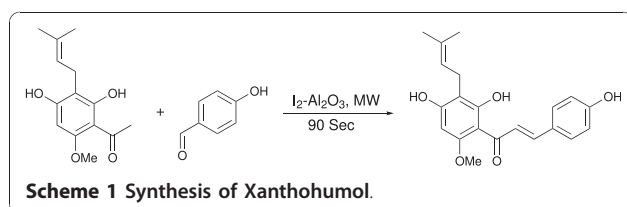
Table 2 Synthetic chalcones prepared using Iodine-alumina under microwave irradiation



Entry	R'	R	Yield ^a (%)
1	H	H	95
2	H	4-OH	93
3	2'-OH	H	91
4	H	3,4-O-CH ₂ -O-	90
5	4'-OH	4-OH	94
6	4'-OMe	4-Cl	88
7	4'-OH	4-OMe	82
8	H	4-NO ₂	82
9	H	3,4-OH	87
10	2'-OH	3-OMe, 4-OH	81
11	3'-OH	2-NO ₂	81
12	4'-OMe	4-OMe	79
13	4'-OH	3,4-OH	86
14	4'-OCH ₂ CH = CH ₂	4-OMe	85
15	2',4'-OMe	4-OMe	89

^a Isolated pure yield.

We further checked the applicability of our method in the synthesis of highly bioactive prenylated hydroxychalcone Xanthohumol. Xanthohumol was isolated in 76% yield in 90 seconds (Scheme 1), with the recovery of unreacted starting which is better than its previously reported method [50].



This new method of preparation of chalcones is particularly attractive since it specifically generates the E isomer from substituted benzaldehyde and acetophenones, a large number of which or their derivatives show one or the other biological activity. Inspection of the ¹H NMR spectra clearly indicates that the chalcones were both geometrically pure and were configured trans.

Experimental Section

All commercially available chemicals and reagents were purchased from Aldrich and used without further purification. Melting points were determined with a Buchi B 540 apparatus in open capillaries and are uncorrected. IR spectra were recorded on a Perkin-Elmer 1640 FT-IR instrument. The ¹H- and ¹³C-NMR spectra were recorded on Bruker DPX-300 NMR machine. Unless

otherwise specified, CD₃OD and CDCl₃ were used as solvent. Mass spectra were recorded with a Trace DSQ GCMS system. Elemental analyses were carried out using a Perkin- Elmer series II CSNS/O Model 2400 analyzer. The I₂-Al₂O₃ catalyst was prepared by the procedure reported earlier [51].

Conclusion

In conclusion, we have developed a new, simple and solvent free method for the synthesis of substituted chalcones using iodine-alumina. The mild reaction conditions, clean reaction profiles, zero side product and cost efficiency render this approach as a useful alternative to the existing methods. Further studies on the application of this method for the synthesis of highly functionalized biologically active chalcones are underway.

Representative procedure

4'-Hydroxy acetophenone (100 mg, 0.735 mmol), 4-hydroxy benzaldehyde (90 mg, 0.735 mmol) and 200 mg 5% I₂-Alumina was taken and homogenized in a mortar. Then the mixture was irradiated in a reaction vessel of a Synthwave 402 Prolabo focused microwave reactor for 80 seconds after setting reaction temperature at 60°C and power at 40% (maximum output 300 W). After cooling to room temperature ethyl acetate (15 mL) was added to the reaction mixture and filtered the mixture through a general laboratory filter paper to separate the solid catalyst. After washing the filtrate with Na₂S₂O₃ solution (1 × 15 mL) and water (1 × 15 mL) the separated organic layer was concentrated under reduced pressure and the product was recrystallized from hot ethanol.

Analytical data of some new compounds as well as melting points of known compounds with their literature values are given below.

1,3-Diphenylpropenone (Entry 1)

Yellow needles. M.P. 56-57°C, Lit. [56-57°C] [34]

3-(4-Hydroxyphenyl)-1-phenylpropenone (Entry 2):

Pale yellow solids. M.P. 181-182°C, Lit. [180-181°C] [30]

1-(2-Hydroxyphenyl)-3-phenylpropenone (Entry 3):

Pale yellow solids. M. P. 88°C, Lit. [88°C] [17]

3-Benzo[1,3]dioxol-5-yl-1-phenyl-propenone (Entry 4):

Fine yellow crystals. M. P. 55°C. IR (KBr) 1660, 1590, 1451, 1310, 1253, 1036, 778. ¹H NMR (300 MHz, CD₃OD): 6.0 (s, 2H, CH₂O₂); 6.9 (d, J = 8.0, 1H, 3-H); 7.25 (dd, J = 1.6, 8.4, 1H, 6-H); 7.4 (d, J = 1.6 Hz, 1H, 1-H); 7.53-7.66 (m, 4H, 3'-H, 5'-H, α-H, 4'-H); 7.7 (d, J = 15.6, 1H, β-H); 8.11-8.10 (m, 2H, 2'-H, 6'-H). ¹³C NMR (75 MHz, CDCl₃):122.07 (O-CH₂-O), 128.49 (C-3, C-6), 128.54 (C-α, C-1), 128.66 (C-3', C-5'), 128.99 (C-2', C-6'), 130.6 (C-1), 132.84 (C-4'), 134.88 (1'), 138.2 (C-β), 144.9 (C-4, C-5), 190.61 (C = O). MS (ESI⁺): m/z 253 [M+H]⁺.

Anal. Calcd. for $C_{16}H_{12}O_3$: C 76.18, H 4.79. Found: C 76.21; H 4.81.

1,3-Bis(4-hydroxyphenyl)propenone (Entry 5):

Yellow solid. M. P. 197-198°C, Lit. [196-198°C] [34]

3-(4-Chlorophenyl)-1-(4-methoxyphenyl)-propenone (Entry 6):

Pale yellow crystals. M. P. 130-131°C, Lit. [130-131°C] [30]

1-(4-Hydroxyphenyl)-3-(4-methoxyphenyl)-propenone (Entry 7):

Pale yellow solid. M. P. 183-184°C, Lit. [184-185°C] [14]

3-(4-Nitrophenyl)-1-phenylpropenone (Entry 8):

Pale yellow solid. M. P. 162°C, Lit. [155-157°C] [52]

3-(3,4-Dihydroxyphenyl)-1-phenylpropenone (Entry 9):

Pale yellow solid. M. P. 200-201°C, Lit. [202-204°C] [31]

3-(4-Hydroxy-3-methoxyphenyl)-1-(2-hydroxyphenyl)-propenone (Entry 10):

Yellow solid. M. P. 155-157°C, Lit. [153-156°C] [31]

1-(3-Hydroxyphenyl)-3-(2-nitrophenyl)-propenone (Entry 11):

Pale yellow solid. M. P. 185-186°C, Lit. [187-189°C] [34]

1,3-Bis-(4-methoxyphenyl)-propenone (Entry 12):

Yellow crystals. M. P. 100°C, Lit. [98-100°C] [52]

3-(3,4-Dihydroxyphenyl)-1-(4-hydroxyphenyl)-propenone (Entry 13):

Pale yellow solid. M. P. 219-220°C, Lit. [218-219°C] [31]

1-(4-Allyloxyphenyl)-3-(4-methoxyphenyl)propenone (Entry 14):

Light yellow crystals. M. P. 76°C. IR (KBr): 2925, 2839, 1658, 1602, 1511, 1254, 1016, 818. 1H NMR (300 MHz, $CDCl_3$): 3.85 (s, 3H, OCH_3), 4.63 (s, 2H, 2''-H), 5.31 (d, $J_{cis} = 10.3$, 1H, 4''-H); 5.42 (d, $J_{trans} = 17.3$, 1H, 4''-H); 6.08 (m, 1H, 3''-H); 6.92-7.0 (m, 4H, 3-H, 5-H, 3'-H, 5'-H); 7.46 (d, $J = 15.6$, 1H, α -H); 7.6 (d, $J = 7.6$, 2H, 2-H, 4-H); 7.8 (d, $J = 15.5$, 1H, β -H); 7.94-8.04 (m, 2H, 2'-H, 6'-H). ^{13}C NMR (75 MHz, $CDCl_3$): 55.42 (OCH_3), 68.91 (2''-C), 114.38 (3-C, 5-C), 114.49 (3'-C, 5'-C), 118.23 (4''-C), 119.49 (C- α), 127.79 (C-1), 130.14 (C-2, C-6), 130.7 (C-2', C-6'), 131.43 (C-1'), 132.55 (3''-C), 143.86 (β -C), 161.51 (4-C), 162.26 (4'-C), 186.76 (C = O). MS (ESI⁺): m/z 295 [M+H]⁺. Anal. Calcd. for $C_{19}H_{18}O_3$: C 77.53, H 6.16. Found: C 77.51; H 6.19.

1-(2,4-Dimethoxy-phenyl)-3-(4-methoxy-phenyl)-propenone: (Entry 15)

Yellow crystals. M. P. 84°C. IR ($CHCl_3$): 2938, 1651, 1573, 1251, 1126, 1026, 828. 1H NMR (300 MHz, $CDCl_3$): 3.85 (s, 3H, OCH_3); 3.87 (s, 3H, OCH_3); 3.9 (s, 3H, OCH_3); 6.49 (d, $J = 2.2$, 1H, 3'-H); 6.54 (dd, $J = 2.2$, 8.6, 1H, 5'-H); 6.9-6.94 (m, 2H, 3-H, 5-H); 7.36 (d, $J =$

15.8, 1H, α -H); 7.53 (d, $J = 9.7$, 2H, 2-H, 6-H); 7.62 (d, $J = 15.7$, 1H, β -H); 7.73 (d, $J = 8.6$, 1H, 6'-H). ^{13}C NMR ($CDCl_3$, 75 MHz): 55.39 (OCH_3), 55.56 (OCH_3), 55.76 (OCH_3), 98.66 (C-3'), 105.07 (C-5'), 114.29 (C-3, C-5), 122.44 (C-1'), 124.98 (C- α), 128.14 (C-1), 130.01 (C-2, C-6), 132.75 (C-6'), 142.11 (C- β), 160.25 (C-4), 161.23 (C-2'), 163.97 (C-4'), 190.71 (C = O). MS (ESI⁺): m/z 298.6 [M+H]⁺. Anal. Calcd. for $C_{18}H_{18}O_4$: C 72.47, H 6.08. Found: C 72.50; H 6.10.

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Authors' contributions

DK carried out the synthetic experiments, purification of compounds, literature survey, spectroscopic analyses and drafted the manuscript. JCS arranged the work and modified the manuscript. Both the authors read and approved the final manuscript.

Competing interests

The authors declare that they have no competing interests.

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