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Short communication

Ultrasonic-assisted synthesis of flavones by oxidative cyclization of 2′-hydroxychalcones using iodine monochloride

Achraf Lahyani, Mahmoud Trabelsi *

Laboratory of Natural Substances, Faculty of Sciences, University of Sfax, Route de Soukra km 3.5, 3038 Sfax, Tunisia

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ABSTRACT

This paper presents an efficient methodology for the synthesis of flavones *via* the oxidative cyclization of 2′-hydroxychalcones in the presence of iodine monochloride with DMSO under ultrasound irradiation. Ultrasonic irradiation enhances the cyclization reaction and leads to reduced reaction time at lower reaction temperatures while generating flavones with high yields.

1. Introduction

Flavones or 2-phenylchromones are abundant in numerous naturally occurring products and constitute an important group of oxygen heterocycles that are widely distributed in the plant kingdom as secondary metabolites [1].

Recently, much attention has been paid to the synthesis of flavones because of their various biological activities, such as anti-inflammatory [2], antiestrogenic [3], antioxidant, anticancer, anti-HIV, anti-hypertensive [4], anti-antimicrobial [5], cardiovascular [6], anti-diabetic [7], anti-allergic [8] and chemo preventative activities [9].

A variety of flavone synthesis methods have been developed. Traditionally flavones have been prepared by Baker–Venkataraman rearrangement [10–12], Allan–Robinson [13], *via* an intermolecular Wittig [14] and Auwers synthesis [15]. Similarly, oxidation of flavanones to flavones is well known in the literature [16,17].

Alternatively, oxidative cyclization of 2′-hydroxychalcones constitutes an important route for the synthesis of flavones, and a number of oxidizing agents such as I_2-DMSO [18], oxalic acid [19], $InBr_3$, and $InCl_3$ [20], $FeCl_3$ [21], I_2-Al_2O_3 [22], Na_2TeO_3 [23], CuI [24], NH_4I [25], DDQ [26] etc. have been reported in the literature for this conversion.

Some synthesis methods are not very satisfactory due to drawbacks such as low yields, high reaction temperature, long reaction time and formation of mixture of product containing flavones, flavanones and aurones have been reported in some cases. Therefore, the development of a new method for efficient synthesis of flavones is strongly desirable.

Iodine monochloride is a versatile reagent for the synthesis of a large number of organic compounds being employed, for example, as a source of electrophilic iodine in the synthesis of mono- or tri-iodinated fluorine aromatic compounds [27]. It can be used as an oxidant to afford 1,3-dioxolan-2-ylium ions from 1,3-dioxolanes [28]. Other examples of synthetic applications of this reagent also include electrophilic cyclizations to obtain various heterocyclic compounds such as 4-iodoisocoumarins [29], 3-iodochromones [30] and 3-substituted-2-chalcogenbenzo[*b*] furans [31].

Ultrasound has increasingly been used in organic synthesis. It is reported that the ultrasound irradiation can lead to the apparent improvement of the reaction efficiency with increased yields and reduced reaction time Ultrasound has increasingly been used in organic synthesis. Studies have shown that ultrasound irradiation can lead to improved reaction efficiency *via* increased yields and reduced reaction time [32]. It is also observed that reactions under ultrasound irradiation are commonly easier to work-up than those in conventional stirring methods [33]. Additionally, in many cases, reactions under ultrasound irradiation represent environmentally friendly processes, using small amounts of solvents and consuming less energy [34]. What's more, ultrasonic irradiation provides minimal side reactions [35]. To the best of our knowledge, there is no report in the literature on the preparation of flavones from 2′-hydroxychalcones using ultrasound irradiation.

* Corresponding author.
E-mail address: Mah.Trabelsi@fss.rnu.tn (M. Trabelsi).

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We purpose in this work an improved synthesis of flavones using an equimolar amount of iodine monochloride with DMSO under ultrasound irradiation. This method allows for high yields in a short time and at low reaction temperatures.

2. Experimental

2.1. Chemicals and apparatus

All solvents and reagents were purchased from Fluka and Sigma–Aldrich and used without further purification. 2′-hydroxy substituted chalcones were prepared by base-catalyzed condensation between 2′-hydroxyacetophenone and the appropriate benzaldehyde using a literature procedure [36]. Wijs' Reagent (0.1 mol/l, 0.2 N) was purchased from Panreac. The ultrasonication was performed in a Bioblock 750 W ultrasound cleaner with a low frequency of 20 kHz (amplitude of 30%). The melting points of the isolated products were measured on a Reichert-Heizbank apparatus. ^{1}H and ^{13}C NMR spectra were acquired in $CDCl_3$ on a Bruker Avance III HD (400 MHz for ^{1}H and 100 MHz for ^{13}C) spectrometer using TMS as internal standard.

2.2. General procedure for the synthesis of flavones

For a typical synthesis, 2′-hydroxychalcones (0.5 mmol) was dissolved in Wijs' reagent (5 mL). The acetic acid was removed in vacuo. Then, the resulting solid and DMSO (5 mL) were charged in a 10 mL glass reactor. The reaction mixture was heated in an oil bath at 50 ± 2 °C. The ultrasound probe was immersed directly in the reactor. The ultrasonic generator (Bioblock Scientific 750 W) emits the sound vibration into the reaction mixture. Sonification was achieved at low frequencies of 20 kHz (amplitude of 30%). The reaction time was fixed at 30 min. When the reaction time was over, the mixture was allowed to cool, poured into 10 mL water and extracted with chloroform (3 × 20 mL). The organic layer was washed with sodium thiosulphate (3 × 10 mL) until neutrality and dried with $MgSO_4$ anhydrous. After filtration, the solvent was removed under reduced pressure to furnish the crude product. The yields of the reactions were calculated from the mass of the isolated pure product.

It is noteworthy that flavones were obtained in excellent yields without the use of any column chromatography. The reactions were clean and isolated compounds provided an NMR pure (>97% purity) product.

2.3. Spectroscopic analysis

In general, no further purification method was required. All the products were previously reported and characterized by the melting point, IR, ^{1}H NMR, ^{13}C NMR.

The spectral data of the isolated compounds, taken as representative examples, are listed below.

2.3.1. 2-Phenyl-4H-chromen-4-one

IR [ν, cm^{-1}] 3088 (C=C–H, Ar–H), 1645 (C=O), 1568 (C=C, Ar), 1128 (COC). ^{1}H NMR [δ, ppm] 8.17 (dd, 1H, J_{H-H} = 1.2 Hz; J_{H-H} = 8 - Hz), 7.85–7.83 (m, 2H), 7.63 (t, 1H, J_{H-H} = 8 Hz), 7.50–7.44 (m, 4H), 7.35 (t, 1H, J_{H-H} = 8 Hz), 6.76 (s, 1H); ^{13}C NMR [δ, ppm] 178.5, 164.4, 156.3, 134.3, 132.1, 131.3, 129.1 (2C), 126.5 (2C), 125.7, 125.6, 123.1, 118.1, 106.8.

2.3.2. 2-(4-chlorophenyl)-4H-chromen-4-one

IR [ν, cm^{-1}] 3086 (C=C–H, Ar–H), 1647 (C=O), 1601, 1568 (C=C, Ar), 1132 (COC). ^{1}H NMR [δ, ppm] 8.23 (dd, 1H, J_{H-H} = 1.6 Hz; J_{H-H} = 8 Hz), 7.88 (d, 2H, J_{H-H} = 8.4 Hz), 7.75–7.71 (m, 1H), 7.58 (d, 1H, J_{H-H} = 8 Hz), 7.50 (d, 2H, J_{H-H} = 8.4 Hz), 7.44 (t,1H, J_{H-H} = 8 Hz), 6.87 (s, 1H); ^{13}C NMR [δ, ppm] 178.1, 162.1, 156.1, 137.8, 133.8, 130.2, 129.3 (2C), 127.5 (2C), 125.7, 125.3, 123.8, 118.0, 107.6.

2.3.3. 2-(4-methoxyphenyl)-4H-chromen-4-one

IR [ν, cm^{-1}] 3051 (C=C–H, Ar–H), 1640 (C=O), 1602, 1572 (C=C, Ar), 1132 (COC). ^{1}H NMR [δ, ppm] 8.22 (dd, 1H, J_{H-H} = 1.2 Hz; J_{H-H} = 8 Hz), 7.88 (d, 2H, J_{H-H} = 8.8 Hz), 7.72–7.67 (m, 1H), 7.55 (d, 1H, J_{H-H} = 8 Hz), 7.41 (m, 1H), 7.01 (d,2H, J_{H-H} = 8.8 Hz), 6.82 (s, 1H), 3.88 (s, 3H); ^{13}C NMR [δ, ppm] 178.2, 165.4, 163.2, 156.2, 134.5, 128.7 (2C), 125.8, 125.6, 123.1, 122.3, 118.0, 114.7, 114.6, 104.7, 55.6.

2.3.4. 2-(3,4-dimethoxyphenyl)-4H-chromen-4-one

IR [ν, cm^{-1}] 3065 (C=C–H, Ar–H), 1696 (C=O), 1606 (C=C, Ar), 1143 (COC). ^{1}H NMR [δ, ppm] 8.23 (dd, 1H, J_{H-H} = 1.6 Hz; J_{H-H} = 8 - Hz), 7.72 (m, 1H), 7.59 (d, 2H, J_{H-H} = 8.8 Hz), 7.44 (m, 1H), 7.40 (d, 1H, J_{H-H} = 2 Hz), 6.99 (d,1H, J_{H-H} = 8.8 Hz), 6.90 (s, 1H), 3.99 (s, 3H), 3.97 (s, 3H); ^{13}C NMR [δ, ppm] 178.3, 163.3, 156.1, 152.0, 149.2, 133.5, 125.6, 125.0, 124.2, 123.8, 119.9, 117.9, 111.1, 108.7, 106.4, 56.0 (2C).

2.3.5. 2-(3,4,5-trimethoxyphenyl)-4H-chromen-4-one

IR [ν, cm^{-1}] 3063 (C=C–H, Ar–H), 1694 (C=O), 1574 (C=C, Ar), 1152 (COC). ^{1}H NMR [δ, ppm] 8.23 (dd, 1H, J_{H-H} = 1.2 Hz; J_{H-H} = 8 Hz), 7.73 (m, 1H), 7.61 (d, 1H, J_{H-H} = 8.4 Hz), 7.44 (t, 1H, J_{H-H} = 7.6 Hz), 7.15 (s, 2H), 6.88 (s,1H), 6.90 (s, 1H), 3.97 (s, 6H), 3.94 (s, 3H); ^{13}C NMR [δ, ppm] 178.4, 163.5, 156.2, 153.5, 141.2, 133.9, 126.8, 125.7, 125.4, 123.6, 118.1 (2C), 107.2, 103.7 (2C), 61.1, 56.3 (2C).

Table 1
Optimization of the DMSO amount.

Entry	Amount of DMSO (mL)	Yield[a,b] (%)
1	0	N.R
2	1	16
3	2	29
4	3	49
5	5	96
6	10	97

[a] Reaction condition: 2′-hydroxychalcone (0.5 mmol), ICl (0.5 mmol), acetic acid (5 mL), refluxed in open air for 60 min at 130 °C.
[b] Isolated yield of flavone.

Scheme 1. Oxidative cyclization of 2′-hydroxychalcone to flavone.

Table 2
Effect of ultrasound irradiation on the reaction yield.

Entry	Time (min)	Temperature (°C)	Yield[a] (%)
1	60	25–30	33
2	60	50	71
3	15	50	71[b]
4	30	50	96[b]
5	30	50	22[b,c]
6	60	50	97[b]

[a] Isolated yield of flavone.
[b] Free of acetic acid.
[c] Without the use of ultrasound.

Table 3
Optimization of the oxidant amount.

Amount of ICl (eq.)	0.2	0.5	0.7	1
Yield[a] (%)	66.7	68.1	76	96

[a] Isolated yield of flavone.

3. Results and discussion

In order to obtain the best cyclization conditions, the transformation of 2′-hydroxychalcone (1a) to 2-phenyl-4H-chromen-4-one (2a) was studied as a model reaction (Scheme 1).

At first, to determine the optimal amount of the DMSO, the model reaction was carried out using different DMSO amounts. The results of this transaction are summarized in Table 1.

As is shown in Table 1, when the reaction was carried out in the absence of DMSO, no product was obtained. At lower amounts of DMSO, the crude ^{1}H NMR spectra of the reaction mixture showed the presence of different products including flavones, in addition to flavanones and starting material (2′-hydroxychalcones); the yield of flavone increased with increasing DMSO amounts. The maximum yield (96%) was obtained by adding 5 mL of DMSO. At amounts higher than 5 mL, the reaction yield remains almost constant.

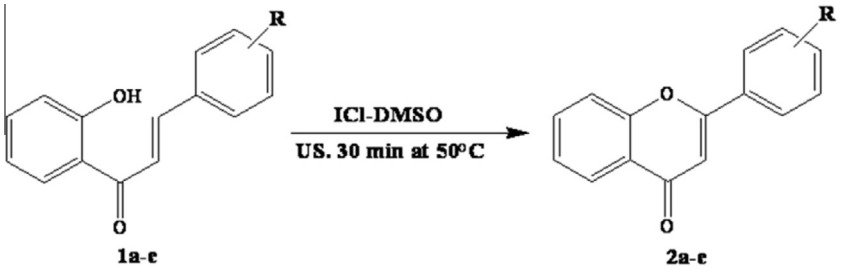
Our next step was to study the effect of ultrasound on % yield, reaction time and temperature reaction in order to understand the role of ultrasound in the synthesis of flavones. The model reaction was carried out under ultrasound irradiation.

As shown in Table 2, increasing the reaction temperature from 25–30 to 50 °C allowed us to obtain the flavone in a moderate yield (71%), after 60 min of ultrasonication. In fact, in the absence of acetic acid, higher flavone yield (97%) is obtained after 60 min of reaction time than the yield obtained when acetic acid is used as solvent (entries 2 and 6). Based on these results, it appears that a temperature of 50 °C is satisfactory for the cyclization reaction of 2′-hydroxychalcone without the use of acetic acid as a solvent.

In order to verify the effect of ultrasound irradiation, we performed the cyclization reaction of 2′-hydroxychalcone by heating at 50 °C for 30 min in the absence of ultrasonic wave. The yield of flavone was 22% (Table 2), which was less than that obtained via ultrasonic-induced synthesis. While under ultrasound irradiation, the reaction could be completed within 30 min in 96% yield at 50 °C. Thus, it was clear from the data that ultrasound can accelerate the cyclization reaction of 2′-hydroxychalcone, affording more yield than thermal conditions and significantly reducing the reaction temperature. In addition, the data in Table 2 (entries 3,4, and 6) show that a fast kinetics reaction was obtained at this temperature. The reaction yield reached 71% after only 15 min and reached its maximum (96.0%) after 30 min of ultrasound irradiation. This finding is due to the phenomenon of cavitation that is grown by reducing the ambient pressure by static or dynamic means under ultrasound irradiation. Ultrasound irradiation activates the reaction mixture by inducing high local temperatures and pressure generated inside the cavitation bubble and its interfaces when it collapses. Ultrasound irradiation also accelerates the reaction rate and shortens the reaction time [37]. Ultrasound procedure therefore presents itself as a green and effective method that can replace the conventional method, because in many reactions the straight heating can disturb the starting materials and the related products and sometimes generate by-products [38].

Next, we investigated the amount of iodine monochloride required to oxidize the 2′-hydroxychalcone. The same model reaction (0.5 mmol of 2′-hydroxychalcone) was carried out using different amounts of iodine monochloride at 50 °C for 30 min under ultrasound irradiation. The results obtained from these reactions are summarized in Table 3.

As is shown in Table 3, the yield of flavone increased with increasing amounts of oxidant. When we increased the amount of iodine monochloride from 0.2 eq. to 0.5 eq., no significant

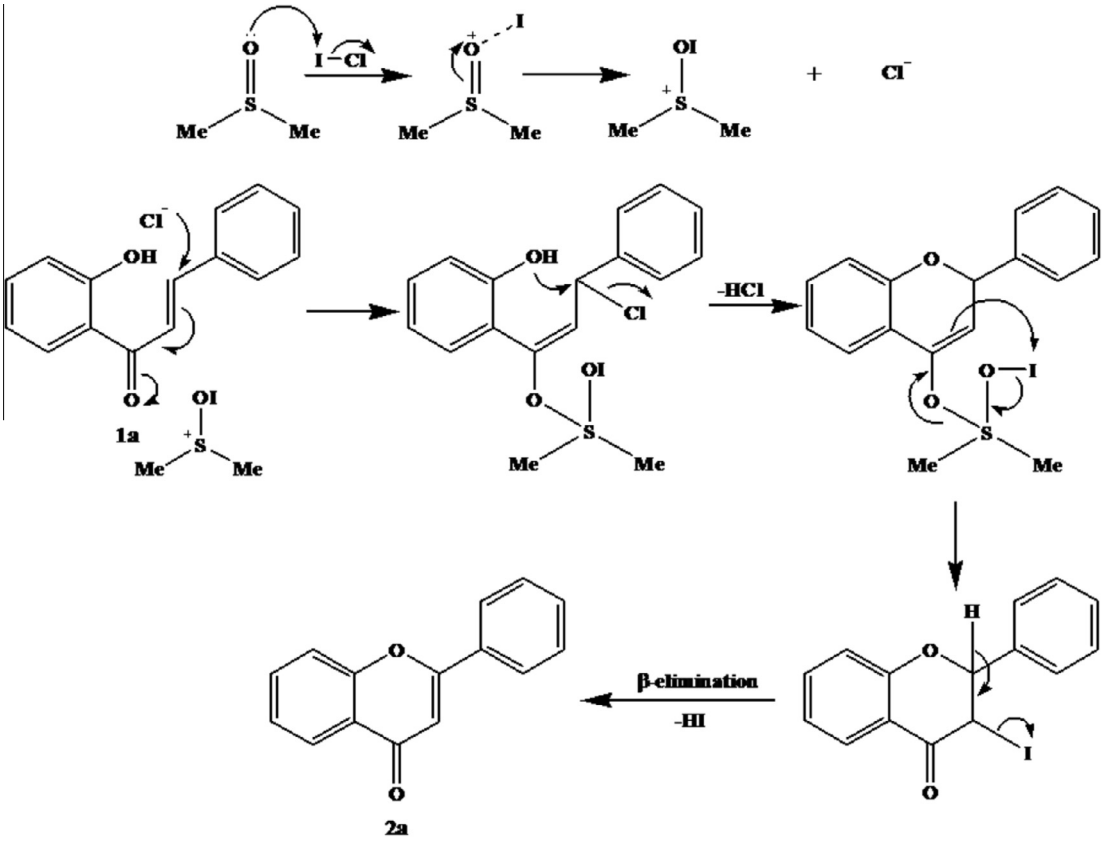


Scheme 2. Synthesis of flavones using ICl-DMSO under ultrasound irradiation.

Table 4
ICl-DMSO mediated oxidative cyclization of 2′-hydroxychalcones under ultrasound irradiation.

Product	Aldehydes	Yield[a] (%)	Melting points (°C)	
			Found	Reported [22,14]
2a	Benzaldehyde	96	98–99	96–97
2b	4-Chlorobenzaldehyde	97	186–187	184–185
2c	4-Methoxybenzaldehyde	96	155–156	158–159
2d	3,4-Dimethoxybenzaldehyde	95	177–178	175–176
2e	3,4,5-Trimethoxybenzaldehyde	93	203–204	204–205

[a] Isolated yield of flavones.



Scheme 3. Proposed mechanism of flavones synthesis using ICl-DMSO *via* oxidative cyclization under ultrasound irradiation.

change in yields was observed (66.7% and 68.1%). Increasing the oxidant amount to 0.7 eq. afforded a product yield of 76%. When increased to 1 eq., the flavone yield was slightly improved to 96%. Therefore, the best results were obtained with the use of stoichiometric amount of iodine monochloride.

To explore the general validity of this process, a series of flavones derivatives were prepared under optimized conditions (Scheme 2): specifically, a temperature of 50 °C, 5 mL of DMSO, and ultrasound irradiation lasting 30 min.

The results of the yields and the melting points of substituted flavones obtained are summarized in Table 4.

The data in this table suggest that electron withdrawing or donating groups do not contribute towards the yield of the reaction, and sufficiently good yields of the desired products are obtained irrespective of the substitution. The reactions were clean, and isolated compounds were obtained in pure form (IR and NMR) without further purification. There was no need to use a column chromatographer to obtain pure products.

We propose the following as a plausible mechanism for the synthesis of flavone *via* the oxidative cyclization of 2′-hydroxychalcone using iodine monochloride with DMSO under ultrasound irradiation [39] (Scheme 3).

The mechanism that we propose demonstrates the important role of DMSO in the oxidative cyclization of 2′-hydroxychalcone (1a) to flavone (2a). Indeed, DMSO did not act only as a solvent but it also activated the iodine chloride reaction.

4. Conclusion

In summary, this paper presents an improved synthesis of flavones by an oxidative cyclization using iodine monochloride with DMSO under ultrasound irradiation. Compared with the conventional methods, the ultrasound procedure provides several advantages, including mild reaction conditions, shorter reaction times, good yields, and environmental friendliness. As a result, we believe this methodology offers an attractive alternative to the use of molecular iodine.

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