

Ultrasound Assisted Facile Synthesis of 2-Benzylidenebenzofuran-3(2H)-ones

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Keywords: *Ultrasound condition, 2-Benzylidenebenzofuran-3(2H)-ones, 1-(2'-Hydroxy-phenyl)-3-phenyl-propenones, Copper Acetate.*

2-Benzylidenebenzofuran-3(2H)-ones commonly known as aurones, are an important class of oxygen heterocyclic compounds of flavonoid family. They exhibit some biological activities such as antioxidant, antifungal, anticancer, enzyme inhibitory, antiparasitic and antileishmanial activities. They are also responsible for imparting yellow color to the flowers and fruits. Owing to their varied importance, a simple and efficient method for the synthesis of 2-benzylidenebenzofuran-3(2H)-ones involving the reaction of 1-(2'-hydroxy-phenyl)-3-phenyl-propenones with copper acetate in ethanol under ultrasonic irradiation conditions has been described. The present method offers a faster reaction and a higher yield than conventional methods.

Introduction

2-Benzylidenebenzofuran-3(2H)-ones commonly known as aurones are an important class of polyphenolic compounds, more specifically, a subclass of the flavonoid family and structurally isomeric to flavones [1,2]. They impart beautiful bright yellow/gold color to flower and fruits [3]. They could be better explored in nature compared to other members of the flavonoid family. More than a hundred differently substituted 2-benzylidenebenzofuran-3(2H)-ones have been identified and characterized to date [2]. However, due to their importance, chemists and biologists are doing much work on their synthesis and

characterization. Geissman and Heaton, in 1943, isolated the first aurone as *anthochlor* pigment naturally from the flowers of *Coreopsis grandiflora* [4]. Apart from flowering plants, the other chief sources of 2-benzylidenebenzofuran-3(2H)-ones are gymnosperms, brown algae, and bryophytes [5]. 2-Benzylidenebenzofuran-3(2H)-ones also used by plants as phytoalexins, which prevent several infections [6].

2-Benzylidenebenzofuran-3(2H)-ones and their derivatives have been found to possess a wide range of biological properties [7,8] such as anti-oxidant [9,10], anti-bacterial [11], anti-diabetic [12], insect antifeedant [13], anti-Alzheimer's [14], tyrosinase inhibition [15], anti-

inflammatory, anti-tumor and anti-cancer [16,17] activities. Some of the important derivatives of 2-benzylidenebenzofuran-3(2*H*)-ones like Sulfuretin, Hemiltron, and Aureusidin, exhibit prohibited iodothyronine deiodinase [18], antiradical activity [19] and known for its DNA strand scission activities [20]. Recently, they have been reported to display a significant antibiofilm and anti-quorum sensing activity [21] and also used to synthesize Dual-State Emissive (DSE) Fluorophores that can be applied in biomedical studies and diagnostic tools [22]. In some cases, they are also known to show more promising and potent biological activities than chalcones and flavones [7].

Though 2-benzylidenebenzofuran-3(2*H*)-ones are scarce but have been synthesized primarily, and two chief routes have been adopted mainly for their synthesis, which include: (i) Oxidative cyclization of 2'-hydroxychalcones and (ii) Condensation of benzofuran-3(2*H*)-ones with aryl aldehydes and various reagents such as Hg(OAc)₂ [23,24], Thallium(III) nitrate [25] and HCl/CH₃COOH [26], KF-Al₂O₃/CH₂Cl₂/MW [27], KOH or NaOH/CH₃OH [28], EDDA/CH₃CN/Ultrasound [29], acetic anhydride [30], deep eutectic solvent [31], Al₂O₃/CH₂Cl₂ [32] have been used for their synthesis respectively. Some other methods, such as cyclization of 2'-acetoxychalcones using tetrabutylammonium tribromide [33], Ring-closing of 2-(1-hydroxy-3-arylprop-2-ynyl)phenols catalyzed by AuCl₂ [34],

dihaloacrylic acids [35] and cyclization of (2-halogenphenyl)(3-phenyloxiran-2-yl)methanones using Cu-catalyst [36] have also been reported for their synthesis.

However, some of the conditions mentioned above have limitations, such as using harsh and hazardous chemicals and reagents, longer reaction time, elevated temperature, poor yields, and formation of side products. Also, as the reaction is being carried out under heating conditions, the appearance of flavones may be accompanied during the response, making the purification of the required 2-benzylidenebenzofuran-3(2*H*)-ones difficult, and these are obtained in meager yields.

These shortcomings led us to develop a safe, environmentally benign, and more efficient method for the synthesis of 2-benzylidenebenzofuran-3(2*H*)-ones. In recent years, ultrasonication has proven to be an important technique and is increasingly being used for the synthesis of organic compounds because it has the advantages of speeding up the diffusion process of solvents and solutes, a simple tool with maximum results and minimum costs compared to conventional methods [37-43].

The ultrasonic method is also a more effective extraction technique for antioxidant materials than the thermal process [39] So, in continuation of our work on the eco-friendly synthesis of organic compounds using ultrasonic irradiation conditions [44], here we have

described an efficient method for the synthesis of 2-benzylidenebenzofuran-3(2*H*)-ones by oxidative cyclization of 1-(2'-Hydroxy-phenyl)-3-phenyl-propenones using Cu(OAc)₂/EtOH under ultrasonic irradiation condition (**Scheme 1**). Ultrasonication improves the cyclization reaction and reduces reaction time at lower temperatures, producing 2-benzylidenebenzofuran-3(2*H*)-ones in high yields.

Experimental Section

Material and methods

Melting points were determined in open capillary tubes. All the chemicals were obtained commercially and used without further purification. The IR spectra were recorded on a Perkin Elmer spectrum BX series FT-IR spectrophotometer with KBr pellets. ¹H NMR spectra were recorded on Bruker Avance 400 MHz instrument using TMS as the internal standard. Sonication was performed using Nessler ultrasound sonicator. 1-(2'-Hydroxy-phenyl)-3-phenyl-propenones required for the present study were prepared using the method available in the literature [45].

General procedure for the synthesis of 2-Benzylidenebenzofuran-3(2*H*)-ones 2a-2j

A mixture of differently substituted 1-(2'-Hydroxy-phenyl)-3-phenyl-propenones (1 mmol), copper acetate (1 mmol), and ethanol (10 ml) was placed under sonication in an ultrasonicator bath for five minutes. The completion of the reaction was monitored by

thin-layer chromatography. The reaction mixture was diluted with ice-cold water and acidified with concentrated HCl. Thus, the yellow solid was filtered, washed with water, and recrystallized from ethanol to give 2-benzylidenebenzofuran-3(2*H*)-ones in high yields.

Spectral data of selected compounds

*2-Benzylidenebenzofuran-3(2*H*)-one 2a:*

IR spectrum (KBr), ν_{\max} , cm⁻¹: 760, 1645, 1690. ¹H NMR spectrum (400 MHz, CDCl₃), δ , ppm (*J*, Hz): 6.85 (s, 1H), 7.10–7.35 (m, 6H), 7.42–7.70 (m, 2H), 7.86–7.90 (d, 1H, *J* = 8.4).

*2-(4-Methoxybenzylidene)benzofuran-3(2*H*)-one 2b:* IR spectrum (KBr), ν_{\max} , cm⁻¹: 755, 1643, 1692. ¹H NMR spectrum (400 MHz, CDCl₃), δ , ppm (*J*, Hz): 3.92 (s, 3H), 6.88 (s, 1H), 7.00–7.25 (d, 2H, *J* = 8.2), 7.35–7.70 (m, 4H), 7.88–7.92 (d, 2H, *J* = 8.2).

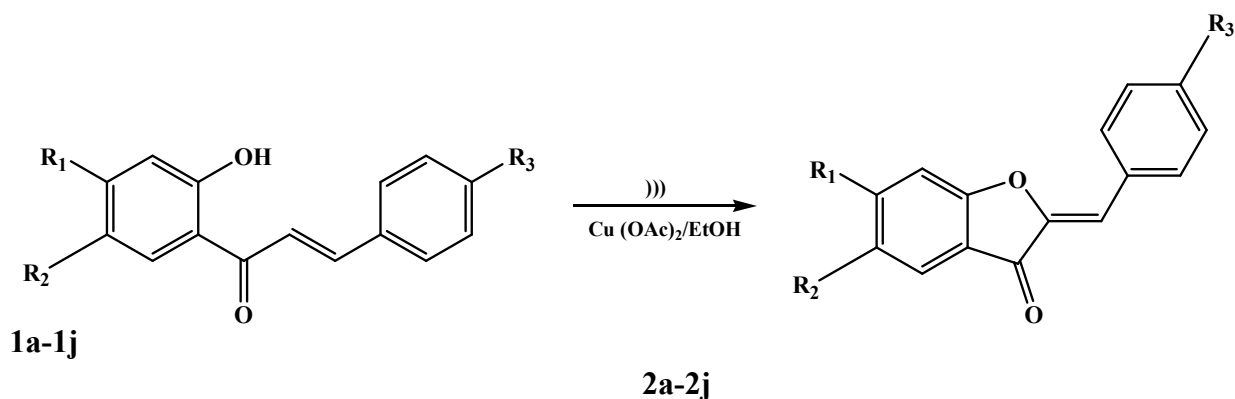
*2-(4-Chlorobenzylidene)benzofuran-3(2*H*)-one 2c:* IR spectrum (KBr), ν_{\max} , cm⁻¹: 750, 1645, 1690. ¹H NMR spectrum (400 MHz, CDCl₃), δ , ppm (*J*, Hz): 6.85 (s, 1H), 6.95–6.98 (d, 2H, *J* = 8.0), 7.28–7.65 (m, 4H), 7.83–7.90 (d, 2H, *J* = 8.0).

*2-(4-Methoxybenzylidene)-5-methylbenzofuran-3(2*H*)-one 2e:* IR spectrum (KBr), ν_{\max} , cm⁻¹: 772, 1650, 1698. ¹H NMR spectrum (400 MHz, CDCl₃), δ , ppm (*J*, Hz): 2.40 (s, 3H), 3.90 (s, 3H), 6.84 (s, 1H), 7.00–7.10 (d, 2H, *J* = 8.2), 7.20–7.26 (d, 1H, *J* = 8.2), 7.40–7.46 (d, 1H, *J* = 8.2), 7.55 (s, 1H), 7.84–7.90 (d, 2H, *J* = 8.2).

2-Benzylidene-6-methoxybenzofuran-3(2H)-one
2g: IR spectrum (KBr), ν_{\max} , cm^{-1} : 762, 1645, 1700. ^1H NMR spectrum (400 MHz, CDCl_3), δ , ppm (J , Hz): 3.92 (s, 3H), 6.82 (s, 1H), 7.10–7.20 (d, 2H, $J=8.2$), 7.38–7.68 (m, 4H), 7.86–7.90 (d, 2H, $J=8.2$).

Results and Discussion

Initially, the reaction of 1-(2'-hydroxyphenyl)-3-phenyl-propenones with $\text{Cu}(\text{OAc})_2$ in ethanol under ultrasonication irradiation condition to afford 2-benzylidenebenzofuran-3(2H)-ones was taken as model reaction (**Scheme 1**). The compound was extracted simply by acidification of the reaction mixture in cold concentrated hydrochloric acid.



Scheme 1. Synthesis of 2-Benzylidenebenzofuran-3(2H)-ones

As the reaction is being carried out under ultrasonic irradiation conditions, it suppresses the formation of flavone as side product thus resulted in the formation of 2-benzylidenebenzofuran-3(2H)-one as sole product. This is confirmed by using thin-layer chromatography. The formation of the above-mentioned product was evidenced by comparison of melting point (100-102 °C) with literature melting point (100-101 °C) [46]. An IR spectrum of the product formed showed absorption at 1690 cm^{-1} , 1645 cm^{-1} and 760 cm^{-1} due to $-\text{C}=\text{O}$, $-\text{C}=\text{CH}$ and $-\text{C}-\text{O}-\text{C}$ stretching respectively and in ^1H -NMR spectrum appearance of a singlet at δ 6.85 due to $=\text{CHPh}$, two multiplets at δ 7.10–7.35 and δ 7.42–7.70 and a doublet at δ 7.86–7.90 due to aromatic protons on Ring A and B revealed that the compound formed was 2-benzylidenebenzofuran-3(2H)-one. Encouraged by these results, variously substituted 2-benzylidenebenzofuran-3(2H)-ones were synthesised by using the above conditions (**Table 1**).

Table 1. Physical data of 2-benzylidenebenzofuran-3(2*H*)-ones synthesised

Compound	R ₁	R ₂	R ₃	Time (min)	Yield ^a (%)	Mp ^b (°C)	Lit. Mp (°C)
2a	H	H	H	5.0	80	100-102	100-101 [46]
2b	H	H	OCH ₃	5.0	88	135-137	137-138 [46]
2c	H	H	Cl	5.0	78	172-174	170 [46]
2d	H	CH ₃	H	5.0	85	115-118	118-119 [46]
2e	H	CH ₃	OCH ₃	5.0	85	151-154	154-155 [46]
2f	H	CH ₃	Cl	5.0	80	183-185	182-183 [46]
2g	OCH ₃	H	H	5.0	80	143-145	142-143 [47]
2h	OCH ₃	H	OCH ₃	5.0	77	132-134	134-135 [47]
2i	OH	H	H	5.0	75	260-262	260-262 [47]
2j	OH	H	OCH ₃	5.0	78	255-256	255-256 [47]

a: Isolated yields; b: melting points are uncorrected and compared with literature values.

Furthermore, to check the present 3(2*H*)-ones is excellent in terms of time and procedure's efficiency, our method's results yield of the product and also avoids the use of were compared with the known methods (Table hazardous and toxic solvents, making the 2). Thus, it is evident that the current protocol for reaction eco-friendly. the synthesis of 2-benzylidenebenzofuran-

Table 2. Comparison of the results of the reactions carried out with different reagents for the synthesis of 2-benzylidenebenzofuran-3(2*H*)-ones with the present method

Sr. No	Catalysts	Time	Temp (°C)	Yield (%)	Ref.
2a	KOH/MeOH/H ₂ O	20 h	Room Temp.	62-72	[28]
2b	HCl/CH ₃ COOH	3-5 h	Room Temp.	62-95	[26]
2c	EDDA/CH ₃ CN	1-5 h	Ultrasound	58-90	[29]
2d	Al ₂ O ₃ /CH ₂ Cl ₂	23 h	Room Temp.	73	[32]
2e	Cu(OAc) ₂ /EtOH*	5 min	Ultrasound	75-88	Present Method

Conclusions

The present protocol for the synthesis of 2-benzylidenebenzofuran-3(2*H*)-ones using Cu(OAc)₂ via oxidative cyclization of 1-(2'-hydroxy-phenyl)-3-phenyl-propenones is highly

efficient and eco-friendly as it avoids the use of toxic organic solvents carried out using ultrasound irradiation condition. This is a clean, mild, highly yielded, and expeditious method that avoids the formation of any by-products.

Acknowledgements

The authors are very much grateful to the authorities of G.D Goenka University, Gurugram, and Kishan Lal Public College, Rewari for their endless support to carry out this research work.

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