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COMMUNICATION

A remarkable solvent effect on the reaction of 4-hydroxycoumarin with (*E*)-3-aryl-2-nitroprop-2-enol: Facile synthesis of highly substituted furo/pyrano[3,2-*c*]chromenes

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A remarkable solvent effect on the reaction of 4-hydroxycoumarin derivatives with (*E*)-3-aryl/hetero-aryl-2-nitroprop-2-enols has been observed in water and DMSO media. This result was employed for the straightforward syntheses of new functionalized furo/pyrano[3,2-*c*]chromenes in 63-93% yields and distereomeric ratio up to ≤99:1. Moreover, a simple, mild, efficient and catalyst-free one-pot method may offer an alternative synthetic strategy for annulating the furan/pyran rings on coumarin nucleus. Furthermore, water has shown a significant positive effect on the rate and selectivity (product) of this reaction.

The development of a highly efficient protocol for the straightforward synthesis of functionalized furocoumarin scaffolds from simple raw materials is a key research area in synthetic organic chemistry and drug discovery programme. A primary reason is that this principal core is frequently found in a broad range of natural products and active pharmacophores,[1] many of them synthetic congeners showing potential biological activities[2] (Figure 1). Owing to the importance of these bioactive furocoumarin derivatives, several efforts have been devoted for the syntheses of substituted 4*H*-furo[3,2-*c*]chromen-4-one derivatives by adopting various modern techniques such as C-O and C-C bond coupling reactions,[3a,3c-d] cascade reaction (Scheme 1a),[3b,3e] lactonization reaction[3g] using transition metal catalysts.[3] However, all these protocols involve toxic/expensive metal catalysts, toxic reagents and harmful/hazardous organic solvents which are not much appreciable from environmental and economic stand points of view.

Besides, the great success of transition metal catalysts, a few examples of metal-free mediated one-pot synthesis of substituted furocoumarin derivatives have been well documented as some of these works were published very recently.[4] For example, in 2012, Feist-Bénary/addition-elimination reaction of 4-hydroxycoumarin with nitroallyl acetate under basic conditions has been realized by Chen and co-workers (Scheme 1b).[4b] Afterwards, in 2013, Wang and co-workers also established a very attractive synthetic strategy involving a four-component reaction between β-nitrostyrene, aromatic aldehyde, 4-hydroxycoumarin and ammonium acetate under refluxing ethanol (Scheme 1c).[4d] Even with a noticeable progress, there is still no successful report on catalyst-free one-pot synthesis of 4*H*-furo[3,2-*c*]chromen-4-one derivatives in water medium. To address this synthetic challenge, we sought to devise an alternative practical, cost-effective and catalyst-free protocol for the preparation of new functionalized furocoumarin scaffolds from simple starting materials under aerobic conditions, specifically, in water which is still a preferred choice of synthetic organic and medicinal chemists.[5]

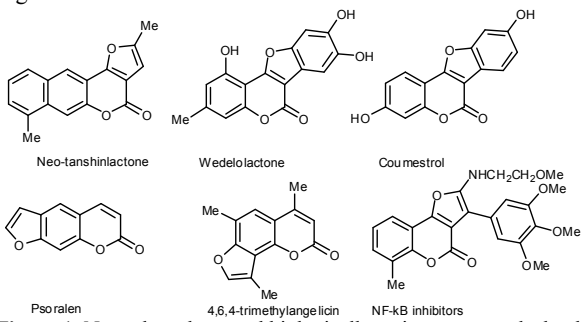
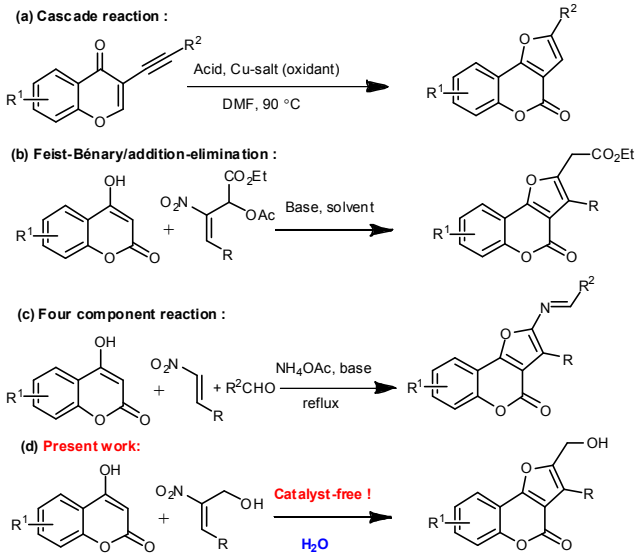


Figure 1 Natural products and biologically active compounds that have a furocoumarin moiety.

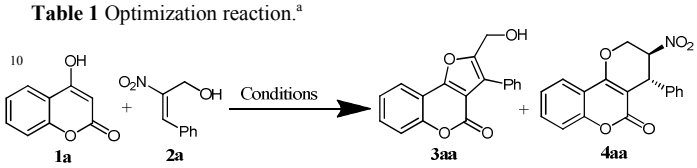


Scheme 1 Various approaches for furo[3,2-*c*]chromen-4-one derivatives.

In recent years, performing the reaction with functionalized β-nitroolefins is an interesting subject for the access of various important heterocyclic scaffolds.[6] In this regard, we also reported

several methods for the syntheses of important class of functionalized heterocyclic compounds utilizing β-nitroolefins as Michael acceptors.[7] Furthermore, we wish to report herein a mild, simple, convenient, high yielding procedure for the construction of 2-(hydroxymethyl)-3-aryl-4*H*-furo[3,2-*c*]chromen-4-one scaffolds via a one-pot reaction of substituted 4-hydroxycoumarins with (*E*)-3-aryl-2-nitroprop-2-enols in water under catalyst-free conditions (Scheme 1d).

Table 1 Optimization reaction.[a]

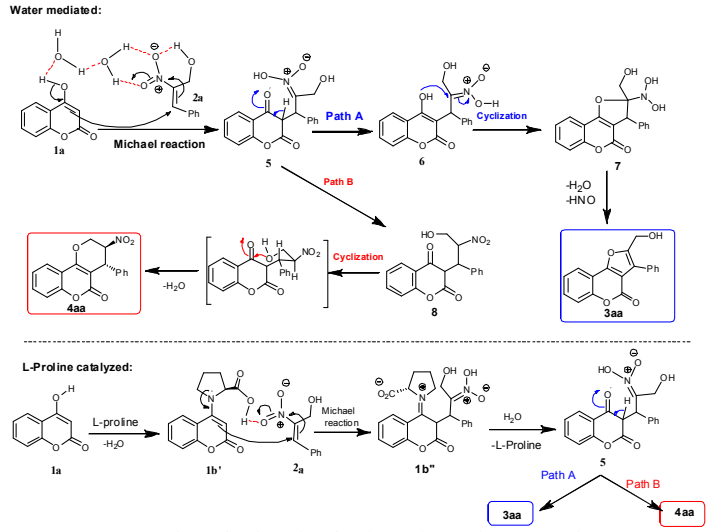


Entry	Catalyst	Solvent	T(°C)	T/h	Yield(%)[c] 3aa	4aa[d]
1[b]	Nil	$CHCl_3$	50	24	12	<3
2[b]	Nil	$CHCl_3$	70	15	25	<5
3[b]	Nil	DCE	70	15	21	<7
4[b]	Nil	Toluene	70	15	41	8
5[b]	Nil	MeCN	70	15	29	<7
6[b]	Nil	EtOH	70	15	19	42
7[b]	Nil	DMSO	70	15	6	85
8[b]	Nil	DMF	70	15	11	81
9	L-proline	$CHCl_3$	70	10	74	9
10	L-proline	DCE	70	10	76	<8
11	L-proline	Toluene	70	10	72	11
12	L-proline	MeCN	70	10	69	7
13	**L-proline**	**DMSO**	**70**	**5**	**7**	**88**
14	L-proline	DMF	70	7	10	83
15	L-proline	EtOH	70	7	29	51
16	DABCO	DMSO	70	5	9	81
17	DMAP	DMSO	70	5	12	83
18	DABCO	DMF	70	7	13	77
19[b]	**Nil**	**H_2O**	**70**	**5**	**83**	**<6**
20	L-proline	H_2O	70	5	86	8
22	DABCO	H_2O	70	5	83	9
23	DMAP	H_2O	70	5	84	<7

[a]Unless otherwise specified, all the reactions were performed with compound **1a** (0.25 mmol), **2a** (0.3 mmol) and catalyst (0.05 mmol, 20 mol%) in specified solvent (0.6 mL) and temperature. [b] Nil indicates no catalyst. [c]Yield of isolated product after column chromatography. [d] Diastereomeric ratio (99:1) of the crude product was recorded by ^{1}H NMR.

The initial reaction between 4-hydroxycoumarin (**1a**) and *E*-3-phenyl-2-nitropro-2-enol (**2a**) was conducted in $CHCl_3$ at 50 °C in the absence of catalyst. After 24h, a trace amount of unexpected 2-(hydroxymethyl)-3-phenyl-4*H*-furo[3,2-*c*]chromen-4-one (**3aa**) along with *trans*-3-nitro-4-phenyl-3,4-dihydropyrano[3,2-*c*]chromen-5(2*H*)-one (**4aa**) were obtained in 12% and 3% yields respectively (entry 1, Table 1). Interestingly, when the same reaction was carried out at elevated temperature 70 °C, slight improvements of results were recorded in terms of reaction time (24h to 15h) and yield (**3aa**, 12% to 25%, entry 2). As we are aware that the reaction of 4-hydroxycoumarin with β-nitrostyrene derivatives highly depends on the polarity of the solvents[8] which necessitates examining the reaction with different solvents in detail. Keeping in mind, we tested this reaction several common organic solvents namely EtOH, DMSO, DMF, $CHCl_3$, 1,2-dichloroethane (DCE), toluene, MeCN at 70 °C. Pleasantly, we noticed that polar solvents like EtOH, DMF and DMSO favoured the formation of **4aa** in good to high yields (42-85%, entries 6-8) and non-polar solvents ($CHCl_3$, DCE, toluene and MeCN) led to the formation of furocoumarin **3aa** in low yields (21-42%, entries 2-5). Screening of several catalysts (L-proline, DABCO, DMAP) revealed that they have almost no influence on product selectivity. However, they enhanced the rate of the reaction (5-10h vs 15h), resulting in higher yields of **3aa** (69-76%, entries 9-12) and **4aa** (51-88% entries 13-18). In order to develop an environmental friendly reaction conditions, we performed this reaction in water instead of harmful organic solvent. Surprisingly, after 5h, in the absence of catalyst, the reaction proceeded very smoothly at 70 °C, leading to the high yield of major product **3aa** (83%, entry 19). In particular, there was no significant improvement of result in terms of yield, selectivity or time when the reaction was carried out in the presence of catalyst (L-proline, DABCO and DMAP) under identical conditions (entries 20-23). From the various reaction conditions as shown in Table 1, it was obvious that best result was obtained for **3aa** at condition mentioned in entry 19 (83% yield).

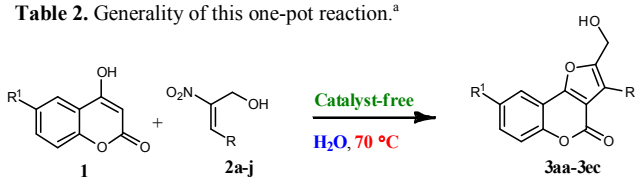
Herein we present the following probable mechanism for the formations of compounds **3aa** and **4aa** under present reaction conditions as shown in Scheme 2. In case of water medium, we assume that water plays a crucial role in this reaction by acting as an amphiphilic dual-catalyst.[9] It may activate both the substrates **1a** and **2a** through intermolecular H-bonding[9a] which increases rate of the Michael addition reaction between **1a** and **2a,** resulting in formation of nitronic acid intermediate **5.** Afterwards, the intermediate **6** is generated from **5** via a tautomerization process which undergoes in turn intramolecular cyclization, subsequent dehydration and elimination of hyponitrous acid (HNO) to give the final compound **3aa** (Path **A**).



Scheme 2 Proposed mechanism for the formations of compounds **3aa** and **4aa.**

On the other hand, the intermediate **5** instead tend to form an intermediate **8** under this condition via a tautomerization process, which is followed by intramolecular cyclization-dehydration, leading to the pyranocoumarin **4aa.** In case of L-proline, we think at this point that Michael reaction may take place through an enamine intermediate **1b'** to form **1b″** which upon reaction with water and followed by elimination of L-proline furnishes intermediate **5.** The finally products **3aa** and **4aa** are generated from **5** by following path **A** and path **B** respectively**.** However, additional work is necessary for understanding the detailed mechanism of this reaction.

Table 2. Generality of this one-pot reaction.[a]



1 **2a-j** **3aa-3ec**

1a; R^1 = H, **1b**; R^1 = Me
1c; R^1 = Cl,**1d**; R^1=Br
1e = NO_2

Entry	R^1	R	T/h	Name	Yield(%)[b]
1	H	Ph	5	**3aa**	83
2	H	4-MeC_6H_4	5	**3ab**	84
3	H	4-$MeOC_6H_4$	5	**3ac**	88
4	H	2-$MeOC_6H_4$	6	**3ad**	87
5	H	3,4-$(MeO)_2C_6H_3$	6	**3ae**	82
6	H	2-HOC_6H_4	10	**3af**	63
7	H	4-ClC_6H_4	5	**3ag**	81
8	H	2-ClC_6H_4	5	**3ah**	85
9	H	4-BrC_6H_4	5	**3ai**	79
10	H	4-$NO_2C_6H_4$	5	**3aj**	83
11	Me	Ph	5	**3ba**	86
12	Me	4-$MeOC_6H_4$	5	**3bc**	88
13	Me	4-ClC_6H_4	5	**3bg**	81
14	Cl	Ph	7	**3ca**	79
15	Cl	4-MeC_6H_4	7	**3cb**	80
16	Cl	4-ClC_6H_4	7	**3cg**	77
17	Br	Ph	7	**3da**	79
18	Br	4-MeC_6H_4	7	**3db**	86
19	Br	4-ClC_6H_4	7	**3dg**	78
20	NO_2	Ph	9	**3ea**	63
21	NO_2	4-$MeOC_6H_4$	9	**3ec**	67

[a]All the reactions were done with 4-hydroxycoumarin derivative (**1a-e**, 0.25 mmol), (*E*)-3-aryl-2-nitropro-2-enols (**2a-j**, 0.3 mmol) in water (0.6 mL) at 70 °C. [b]Isolated yield of after column chromatography.

With the optimum reaction conditions in hand, various substituted (*E*)-3-aryl-substituted-2-nitroprop-2-enols and 4-hydroxycoumarin derivatives were examined to understand the generality and scope of this reaction. The outcomes are compiled in Table 2. It is noteworthy that both electron-donating (Me, MeO and OH) and electron-withdrawing (Cl, Br, and NO_2) groups on the aromatic rings of 3-aryl-substitited-2-nitroprop-2-enols annulated smoothly with substrate (**1a**), providing the functionalized furo[3,2-*c*]chromen-4-ones (**3aa-3aj**) in good to high yields (63-88%, entries 2-10). Similarly, incorporation of several functional groups such as electron donating (Me) and electron withdrawing (Cl, Br and NO_2) on aryl rings of 4-hydroxycoumarins (**1b-e**, entries 11-21) did not pose any with (*E*)-3-aryl-substituted-2-nitroprop-2-enols (**2a-c** and **2g**, entries 11-21) by this procedure and resulted in clean and complete Michael/cyclization-elimination reactions, providing the corresponding annulated products (**3ba-3ec**) in good to high yields (63-88%). It is observed that 4-hydroxycoumarins (**1c-e**) possessing electron-withdrawing substituents are slightly less reactive than its un-substituted version **1a** towards Michael acceptors under identical conditions (e.g. 5h vs 9h, entry 1 vs entry 20). Importantly, our present conditions are mild enough to retain various functional groups such as OMe, OH, Cl, Br, NO_2, CH_2OH etc.

Besides, the gram scale preparation of compound **3aa** was investigated in our imposed conditions. A heterogeneous mixture of compound **1a** (1.62 gm, 10.0 mmol) and **2a** (12.0 mmol) in water (6.0 mL) was heated at 70 °C for 6h. Afterwards, water was decanted carefully from the reaction mixture to give the gummy residue which was directly purified by column chromatography technique, leading to the pure product **3aa** in 76% yield. This interesting result reveals that our optimal condition can be used for milligram to gram scale synthesis.

Next, we turned our attention towards the facile synthesis of functionalized pyranocoumarin derivative as this motif is frequently existed in a variety of bioactive natural products and pharmacophores.[10,11] Literature survey shows that several practical and efficient techniques are available for the syntheses of both the racemic and enantio-enriched versions of dihydropyrano[3,2-*c*]chromen derivatives.[11] Towards our investigations, various functionalized 4-hydroxycoumarin derivatives (**1a-b, 1e**) were reacted with several aryl/hetero-aryl-substituted nitroallylic alcohols in DMSO medium at 70 °C in the presence of L-proline (20 mol%). The results are summarized in Scheme 3. To our delight, all the reactions proceeded smoothly by this procedure to furnish the corresponding previously unknown class of substituted *trans*-3-nitro-4-aryl-3,4-dihydropyrano[3,2-*c*]chromen-5(2*H*)-one derivatives in high to excellent yields (85-93%, **4aa-4ec**) with excellent diastereoselectivities (up to ≤99:1 *dr*). It should be noted that relative stereochemistry of major diastereomer **4ah** was unanimously confirmed by its single crystal X-ray diffraction data (Figure 2, details in ESI) indicating aryl group in *trans*-orientation with NO_2. Similarly, several sensitive functional groups namely NO_2, OMe, furan, Me and Cl are tolerable in our present conditions.

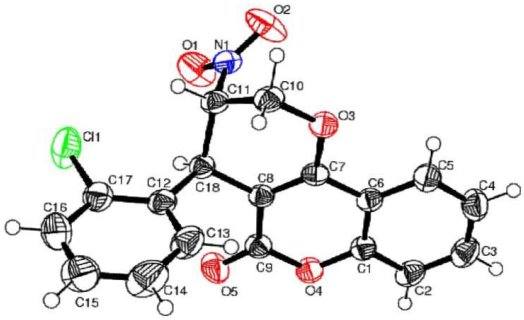
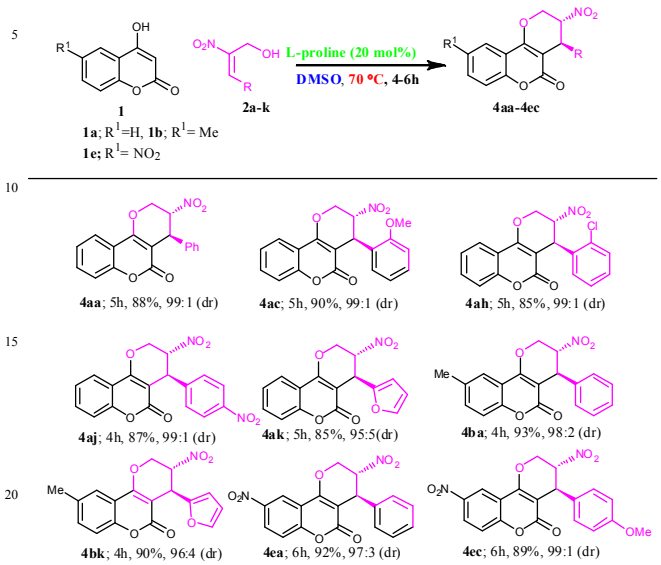


Figure 2 ORTEP diagram of major diastereomer **4ah**, thermal ellipsoids drawn at the 50% probability level.



Scheme 3 One-pot synthesis of *trans*-3-nitro-4-aryl-3,4-dihydropyrano[3,2-*c*]chromen-5(2*H*)-ones.

In conclusion, we have observed a remarkable solvent effect on one-pot reaction of 4-hydroxycoumarin derivatives with (*E*)-3-aryl-substituted-2-nitroprop-2-enols and it was used for expeditious synthesis of highly substituted furo/pyrano[3,2-*c*]chromenes, by choosing water or DMSO as a solvent. Moreover, water has shown not only positive effects on the rate and selectivity (product) of this reaction but also beneficial features in terms of safety, health, cost-effectiveness and environmental points of view**.** Furthermore, our current procedure avoids the use of toxic metals and their salts, harmful/volatile organic solvents, expensive reagents, any need for dry conditions or inert-atmosphere, multi-step, etc. Importantly, in comparison to the established methods, our protocols are advantageous since they are operationally simple, easy to work-up, applicable for gram-scale synthesis, as well as offer good to excellent yields (63-93%), excellent diastereoselectivities (up to ≤ 99:1 dr) and good substrate scope. Further endeavours towards the detailed understanding of the reaction mechanism as well as applications of these compounds are under investigation and will be communicated in due course.

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A remarkable solvent effect on the reaction of 4-hydroxycoumarin with (*E*)-3-aryl-2-nitroprop-2-enol: Facile synthesis of highly substituted furo/pyrano[3,2-*c*]chromenes

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The solvent dependent reaction of 4-hydroxycoumarin with (*E*)-3-aryl-2-nitroprop-2-enol has been reported for the synthesis functionalized furo/pyrano[3,2-*c*]chromenes in water and DMSO media.