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$SnCl_2{\cdot}2H_2O$ catalyzed one-pot three components synthesis of pyrano[4,3-b]chromenes and chromeno[4,3-b]chromenes

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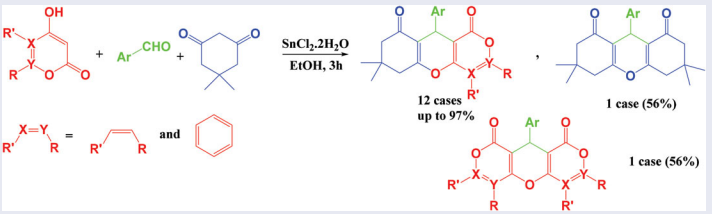
ABSTRACT

A new green method has been presented for the synthesis of some novel derivatives pyrano[4,3-b]chromenes and chromeno[4,3-b]chromenes based on bio-mass triacetic acid lactone or 4-hydroxycoumarine via a one-pot three components condensation reaction of triacetic acid lactone, dimedone, and an aromatic aldehyde using $SnCl_2.2H_2O$ as a catalyst in ethanol at 60 °C. Several activated and deactivated aromatic aldehydes were selected to afford the products in high to excellent yields. Advantages of these syntheses are operational simplicity, mild reaction conditions, environmentally friendly and easy workup.

GRAPHICAL ABSTRACT



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[43-b]Chromene; 4-hydroxycoumarine; multi-component reaction; triacetic acid lactone; tin (II) chloride

Introduction

One-pot multicomponent reactions (MCRs) are very important in biological, medicinal chemistry and modern organic synthesis.[1–4] MCRs represent various advantages than classical multi-steps reactions such as facility, high yield synthesis with simple starting materials, decrease of cost, energy, waste production and time reaction.[5,6]

From of the most important heterocyclic groups are fused chromenes derivatives such as pyrano[4,3-b]chromene and chromeno[4,3-b]chromenes. Studies have shown they have biological and medicinal properties such as cancer therapy,[7] antifungal,[8] antioxidant,[9,10] antiviral,[11,12] anti-microbial,[13,14] and insecticidal.[15] These compounds are present in the nature; many derivatives of them could be synthesized in laboratories by adding different groups into their main heterocyclic structures.[16,17] Triacetic acid lactone (TAL) and 4-hydroxycoumarine (HOC) can be used respectively

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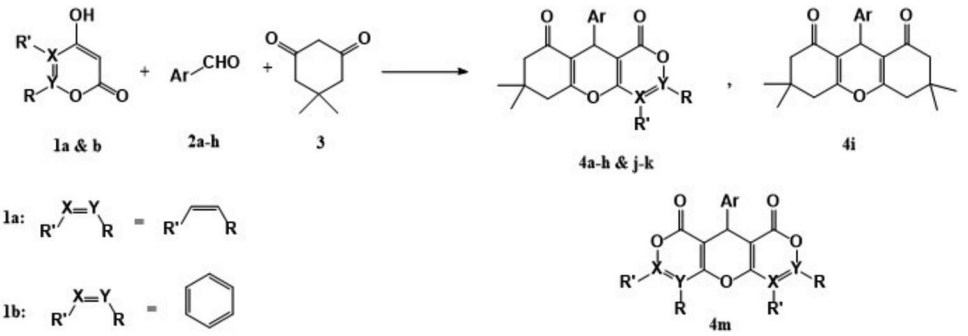


Scheme 1. Reaction of 4-hydroxy 2-pyridones, an aromatic aldehyde and dimedone.

Table 1. Synthesis of **4a** in different catalysts and solvents[a].

Entry	Solvent (mL)	Catalyst (mol%)	Yield (%)[b]
1	EtOH (1)	p-TSA (50)	28
2	EtOH (1)	TEA (50)	5
3	EtOH (1)	–	10
4	EtOH (1)	$SnCl_2.2H_2O$ (50)	63
5	EtOH (1)	$CoCl_2.H_2O$ (50)	30
6	EtOH (1)	$CdCl_2.H_2O$ (50)	51
7	EtOH (1)	$ZnCl_2.H_2O$ (50)	26
9	neat	$SnCl_2.2H_2O$ (50)	50
10	H_2O (1)	$SnCl_2.2H_2O$ (50)	25
11	DMF (1)	$SnCl_2.2H_2O$ (50)	10

[a]Reaction conditions: TAL (1 mmol), Benzaldehyde (1 mmol), dimedone (1 mmol) at reflux in 18 hours.
[b]Isolated yield

for the synthesis of biological effective pyrano[4,3-b] chromene and chromeno[4,3-b]chromenes derivatives.[18,19] ZnO[20] nanoparticles, $Mg(ClO_4)_2$,[21] molybdic acid-magnetic nanoparticles,[22] *p*-toluene sulfonic acid[23] and so have been formally applied in MCRs for synthesis of these compounds. Although, some of these methods are effective, but some of them have inappropriate factors such as long reaction time, hard performance and workup, high-temperature conditions, and expensive reagents.

Bio-mass TAL and HOC as a 2-pyrone compound is one of the natural byproduct of polyketide origin,[24,25] has been applied as precursors in the synthesis compounds. Its derivatives exhibit various biological activities, such as antimicrobial,[26,27] anticoagulant agent,[28–34] antifungal,[35] and antiparasitic activities.[36,37]

Due to the biological properties of these compounds, also in continuing of research plan on the synthesis of important heterocyclic compounds,[38] herein we report a synthetic method for the producing of derivatives of pyrano[4,3-b]chromene and chromeno[4,3-b]chromenes in green condition reaction.

Results and discussion

We initially performed the reaction between TAL, benzaldehyde and dimedone in ethanol in the presence of p-TSA (50 mol%) as a catalyst in the reflux condition (Scheme 1). This reaction produced **4a** (Entry 1, Table 1) in low yield (28%). In the next steps, the reaction was studied under various conditions to afford optimal conditions as

Table 2. Synthesis of **4a** in different amounts of $SnCl_2{\cdot}2H_2O$[a].

Entry	Catalyst (mol%)	Yield (%)[b]
1	75	40
2	50	63
3	30	67
4	20	79
5	10	79
6	5	71

[a]Reaction conditions: TAL (1 mmol), Benzaldehyde (1 mmol), dimedone (1 mmol) at reflux in ethanol (1 mL) for 18 hours.
[b]Isolated yield.

TABLE 3. Synthesis of **4a** in different times and temperature[a].

Entry	Temperature (°C)	Time (h)	Yield (%)[b]
1	Reflux	18	82
2	70	18	85
3	60	18	88
4	50	18	75
5	60	9	88
6	60	4	88
7	60	3	88
8	60	2	50

[a]Reaction conditions: TAL (1 mmol), benzaldehyde (1 mmol), dimedone (1 mmol) and $SnCl_2{\cdot}2H_2O$ (10 mol%) in ethanol (1 mL).
[b]Isolated yield.

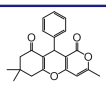
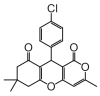
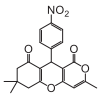
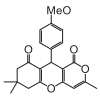
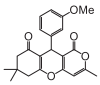
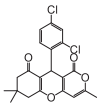
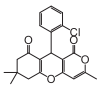
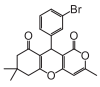
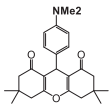
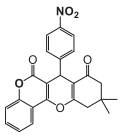
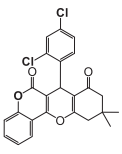
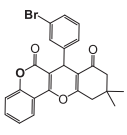
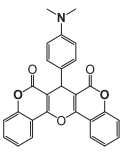
follows: different catalysts (acidic, basic and without catalyst) (Table 1), solvents (Table 1), amounts of catalyst (Table 2), reaction times and temperatures (Table 3). The best yield of product (88%) was obtained in the presence of $SnCl_2.2H_2O$ (10 mol%) in ethanol at 60 °C temperature (Table 3, entry 7). This condition for the reaction was simple, resulting in an economic process, which has clear advantages as an environmentally friendly, in organic synthesis.

Several activated and deactivated aromatic aldehydes be subjected to the reaction with TAL and HOC to give the corresponding **4a–m** in high to excellent yields (Table 4.). In the case of 4-(dimethylamino) benzaldehyde unlike other benzaldehydes, this type product was not separable under this condition and the only products **4i** or **4m** were separated corresponding, from the reaction of two molecules of dimedone with one molecule of aldehyde or two molecules of HOC with one molecule of aldehyde (Table 4, Entry 10 & 14).

^{1}H-NMR spectra of products show that the two methylenic hydrogens of dimedone ring, linked to double bond, are diastereotopic and appear as two doublets (as a sample for **4a** δ: 2.28 and 2.11 ppm, $J = 18$ Hz; Fig. 1a). This is due to the rigidity of structures that causes two hydrogens placed in different chemical media (Fig. 1a,b). Also, this trend was observed in two $-CH_3$ on dimedone (**4a** δ: 0.93 and 1.04 ppm; Fig. 1a). Other methylene of dimedone ring and $-CH_3$ of TAL were observed as two singlets (**4a** δ: 2.60 and 2.18 ppm, Fig. 1a). The ^{13}C NMR spectra of products were adoptable with their structures, but the peak CH pyran ring is located below DMSO peaks, so they are hidden (except in **4a** and **4f** Fig. 1c).

A suggestion mechanism for the formation of **4a** is shown in Scheme 2. It is reasonable to imagine that initially TAL and aldehyde condense and produce **5**, and then **5**

Table 4. Synthesis of derivatives pyrano and chromeno[4,3-b]chromene by $SnCl_2{\cdot}2H_2O$[a].

				mp °C	
Entry	Product	Product	Yield (%)[b]	Found	Lit.
1		4a	88	219–220	–
2		4b	96	264–266	266–268 [21]
3		4c	97	242–244	245–247 [21]
4		4d	87	228–230	229–230 [24]
5		4e	86	185–187	–
6		4f	85	182–184	–
7		4g	83	180–182	–
8		4h	91	215–216	–
10		4i	56	192–194	–
11		4j	87	219–220	220–223 [24]
12		4k	80	250–252	207–208 [22]
13		4l	83	257–259	258–260 [22]
14		4m	51	192–194	–

[a] Isolated yield

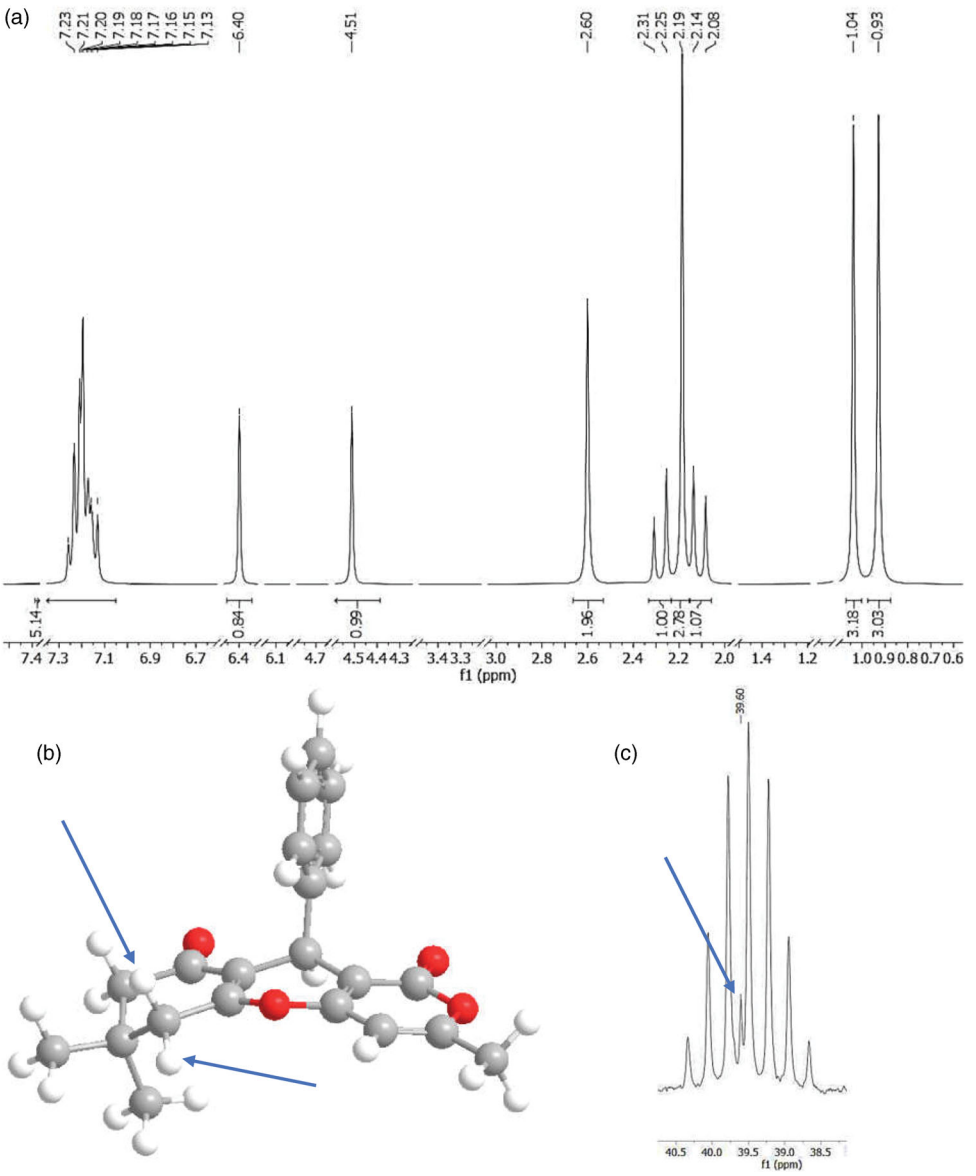
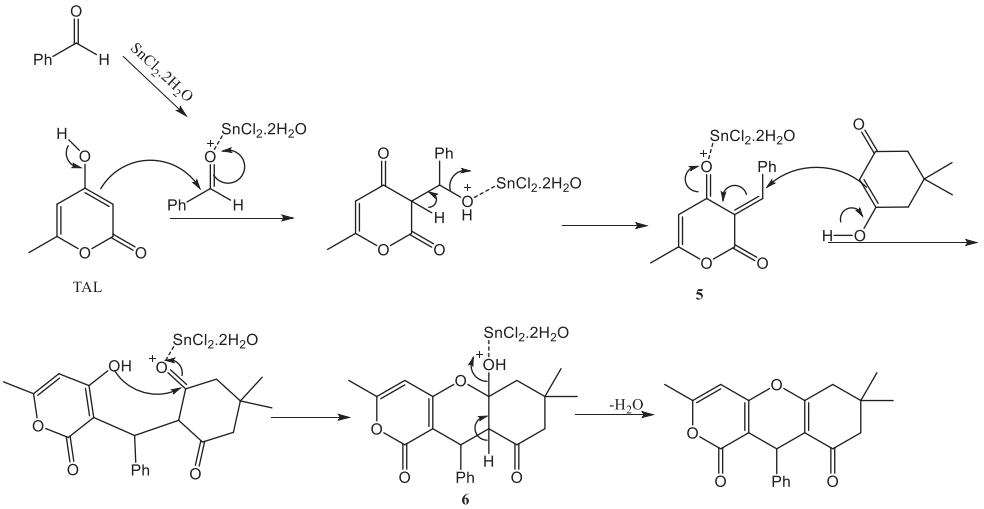


Figure 1. (a) ^{1}HNMR spectrum of **4a.** (b) stereo structure of **4a**. (c) ^{13}C NMR peaks of DMSO region **4a**.

and dimedone react via Michael addition reaction to afford hemiketal **6** which through a dehydration generate **4**.

Experimental section

The starting materials were purchased from Merck and Fluka chemical companies. Melting points were determined with a Branstead Electrothermal model 9200 apparatus and are uncorrected. IR spectra were recorded on a Perkin Elmer RX1 Fourier



Scheme 2. Suggestion mechanism for synthesis of compound **4a**.

transform infrared spectrometer. The ^{1}H and ^{13}C NMR spectra were recorded in DMSO-d_6 on Bruker Avance 300 MHz spectrometers. Elemental analyses were carried out by a Perkin Elmer 2400 series II CHN/O analyzer.

General procedure of synthesis of 4a–i

Triacetic acid lactone (1 mmol), an appropriate aromatic aldehyde (1 mmol) and dimedone (1 mmol) were added to a solution of $SnCl_2{\cdot}2H_2O$ (10 mol%) in absolute ethanol (1 mL) and the mixture was stirred at 60 °C for 3 h. Then the reaction mixture was poured in ice water (10 mL) and the precipitated was collected by filtration, washed with distilled water (10 mL), and dried. The crude product was recrystallized from ethanol (80%) (5 mL) to give the corresponding pure product (**4a–i**).

Spectral data for 4a (as a sample):

White solid, yield 88%, mp 219–220 °C; IR (KBr) ν: 2958 (C–H), 1716 (C = O), 1664 (C = O), 1366 (C–O) cm^{-h}; ^{1}H NMR (300 MHz, DMSO-d_6) δ: 7.13 – 7.26 (m, 5H, Ar-H), 6.40 (s, 1H, =C–H of TAL), 4.51 (s, 1H, CH of pyran), 2.60 (s, 2H, CH_2 of dimedone), 2.28 (d, J = 16 Hz, 1H, =C-CH_2 of dimedone), 2.18 (s, 3H, CH_3 of TAL), 2.11 (d, J = 16 Hz, 1H, =C–CH_2 of dimedone), 1.04 (s, 3H, –CH_3 of dimedone), 0.93 (s, 3H, –CH_3 of dimedone); ^{13}C NMR (75 MHz, DMSO-d_6) δ: 195.9 (C = O dimedone), 162.8 (=C–O pyran), 162.4 (C = O TAL), 161.8 (=C–O TAL), 158.4 (=C of dimedone), 143.2 (C of Ar-H), 128.2 (CH of Ar-H), 128.0 (CH of Ar-H), 126.6 (CH of Ar-H), 113.8 (=C of dimedone), 102.6 (=CH of TAL), 98.2 (=C of TAL), 49.9 (CH_2 of dimedone), 39.6 (C of pyrane), 32.3 (CH_2 of dimedone), 31.9 (C of dimedone), 28.5 (CH_3 of dimedone), 26.6 (CH_3 of dimedone), 19.3 (CH_3 of TAL); Anal. Calcd for $C_{21}H_{20}O_4$: C, 74.98; H, 5.99; O, 19.02. Found: C, 74.91; H, 5.93.

Conclusion

In conclusion, we have successfully developed a quick, convenient, and efficient method for the synthesis of Pyrano[4,3-b]chromene derivatives via a three component condensation using $SnCl_2.2H_2O$ as inexpensive catalyst. The environmentally advantages of this reaction include generality and simplicity of procedure, short reaction time, simple workup, and high to excellent yields.

The experimental details including general procedures, characterization data, copies of ^1H & ^{13}CNMR and FT-IR spectra are available in the supporting information.

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