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Method Article

$Zr(HSO_4)_4$: Green, efficient and reusable catalyst for one-pot synthesis of 1,8-dioxooctahydroxanthene under solvent-free conditions

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A B S T R A C T

Many methods have been used to synthesize xanthene derivatives using different catalysts. However, some of these methodologies have not been entirely satisfactory. Most of the mentioned methods have disadvantages such as low yields, prolonged reaction times, harsh reaction conditions and the requirement of expensive catalysis and use of toxic organic solvent. In this research, a green and highly efficient procedure for the one-pot synthesis of 1,8-dioxo-octahydro-xanthenes has been developed. $Zr(HSO_4)_4$ catalyst was used as an efficient and recoverable catalyst for synthesis of 1,8-dioxo-octahydro-xanthene derivatives *via* cyclocondensation of dimedone and aromatic aldehydes in solvent-free conditions. There are no examples of the use of $Zr(HSO_4)_4$ for the synthesis of 1,8-dioxo-octahydro-xanthene derivatives. The present method offers several advantages such as green, highly efficient, recoverable, reusable, simple work-up and simple purification of products. The structure of the synthesized products was confirmed by Fourier Transform Infrared (Ft-IR) and Proton nuclear magnetic resonance (^{1}HNMR) analyzes. The antibacterial activity of the synthesized compounds was determined by agar disk diffusion method against gram-positive (S. aureus bacteria) and gram-negative (E. coli bacteria) microorganisms. Among the synthesized compounds (3a-3j), 3h compound showed the highest antibacterial effect by forming an inhibitory diameter zone of 15 mm around the disc containing 2000 mg of 3h-compound against gram-positive (S. aureus bacteria).

1. Use of $Zr(HSO_4)_4$ as a green and highly efficient and reusable heterogeneous catalyst.
2. Under solvent-free condition.
3. Simple work-up and Simple purification of products.

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Method details

Introduction

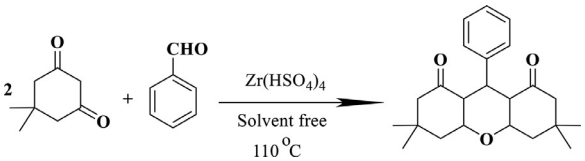
In recent years, synthetic chemists have shown tremendous interested in developing highly efficient transformation for the synthesis of xanthene derivatives due to their potential applications in the biological activities, pharmaceutical and industrial applications. These compounds have antibacterial properties due to the presence of reactive oxygen in their structure. For example, they have many biological and therapeutic properties such as anti-inflammatory [1], antiviral [2], antibacterial [3], as well as in photodynamic therapy [4] and as antagonists of the paralyzing action of zoxazolamine [5]. Xanthenes are also available from natural sources [6]. Many procedures are disclosed to synthesize xanthenes and benzoxanthenes such as montmorillonite K10 [7], nano-$ZnAl_2O_4$ [8], sodium hydrogen sulfate [9], cyclodehydrations [10], [C_4dabco][BF_4] ionic liquid [11], $Smcl_3$ [12], acid activated clay [13], sulfonic acid functionalized silica [14], template-containing Zn/MCM-41 [15], Sulfamic acid [16], Nickel-Cobalt Ferrite [17], Many different methods have been reported for the synthesis of xanthenes; one of them is the condensation of aldehydes with cyclohexane-1,3-dione or 5,5-dimethylcyclohexane-1,3-dione to give 1,8-dioxooctahydroxanthene derivatives. This reaction has been conducted in the presence of strong protonic acids [18], Lewis acids such as $InCl_3.4H_2O$ [19], $FeCl_3.6H_2O$ [20] and heterogeneous catalysts like Dowex-50W [21], $NaHSO_4.SiO_2$ [22], Ambertyst-15 [23], HBF_4/SiO_2 [24] and Sulfonic acid-functionalized LUS-1 [25]. Other catalysts such as alum-promoted [26], potash alum [27], boric acid [28], $ZrOCl_2.8H_2O$ [29], nano-Fe_3O_4 [30], $B(HSO_4)_3$ [31], TiO_2 [32], nano-TiO_2 [33], $BiVO_4$-NPs [34] and $InCl_3$ [35] and etc. [36–38].

However, some of these methodologies have not been entirely satisfactory. Most of the mentioned methods have disadvantages such as low yields, prolonged reaction times, harsh reaction conditions and the requirement of expensive catalysis and use of toxic organic solvent. Therefore, the use of Zr (HSO4) 4 as a heterogeneous catalyst has been considered in this research to avoid these limitations and to create an alternative pathway for the synthesis of xanthene derivatives. $Zr(HSO_4)_4$ is a low-cost solid Brønsted acid. There are no examples of the use of $Zr(HSO_4)_4$ for the synthesis of 1,8-dioxooctahydroxanthene derivatives. In this research, $Zr(HSO_4)_4$ catalyst was used as an efficient, recoverable and reusable catalyst for synthesis of 1,8-dioxooctahydroxanthene derivatives *via* traditional route from cyclocondensation of dimedone and aromatic aldehydes in solvent-free conditions.

Experimental

All chemicals used were of synthetic grade and were used as received without any further purification. Melting points were determined in open glass capillaries on an Electrothermal 9100s apparatus and are uncorrected.

Table 1
Screening of the catalyst and solvent for the reaction of benzaldehydes and dimedone catalyzed by $Zr(HSO_4)_4$.[a]



Entry	Catalyst (mol%)	Solvent/condition	Time (min)	Yield[b] (%)
1	20	water/reflux	120	trace
2	20	THF/reflux	120	trace
3	20	$CHCl_3$/reflux	120	20
4	20	CH_3CN/reflux	120	15
5	20	CH_3CH_2OH/reflux	100	55
6	10	solvent-free	100	68
7	20	solvent-free	40	86
8	30	solvent-free	40	75
9	0	solvent-free	40	trace

[a] Reaction condition: dimedone (2 mmol), aldehyde (1 mmol) and $Zr(HSO_4)_4$ as catalyst.
[b] Isolated yield.

The IR spectra were recorded with FT-IR Shimadzu IR-470 instrument using potassium bromide pellets. The ^{1}H-NMR spectra were determined on a Bruker Avance DRX-400 MHz instrument using TMS as the internal standard and $CDCl_3$ as solvent. Chemical shifts are expressed as δ(ppm) and the coupling constant as $J(H_2)$. The progress of reaction was monitored by Thin-layer chromatography (TLC) using 0.2 mm Merck silicagel GF254 pre-coated plates and visualized by UV-light (254 nm).

Scheme 1

General procedure for the preparation of 1,8-dioxooctahydroxanthene **(3a-j)**: To a mixture of dimedone 1 (2 mmol) and aldehyde 2 (1 mmol) was added $Zr(HSO_4)_4$ which prepared as reported [1] (20 mol%) and the mixture was allowed to stir at 110 °C for the total recorded time which indicated in Table 1. The progress of reactions was monitored by TLC (eluent: EtOAc/ Diethylether, 1:4). After completion of the reaction, the reaction mixture was cooled to room temperature. Then to mixture was added cold water and the product was extracted with ethyl acetate (3 × 5 mL). The organic layer was dried ($MgSO_4$) and evaporated in vacuum. The crude product recrystallized by EtOH 96% and purified. All the desired products were characterized by comparison of their physical data with those reported compounds. For the total confirmed of synthetic compounds, the spectral data given below.

3,3,6,6-tetramethyl-9-phenyl-1,8-dioxooctahydroxanthene **(3a)**: White powder, Yield (86%), mp 201-202 °C. FT-IR ($\bar{\upsilon}$, Cm^{-1}) (KBr disc): 3040 (CH_{arom}, Str.); 2990 (CH_{aliph}, Str.); 1605 (C=O Str.); 1540 (C=C Str.); 1360 (C-O Str.). ^{1}H NMR (400 MHz, $CDCl_3$) δ(ppm): 1.12 (6H, s, $2CH_3$); 1.26 (6H, s, $2CH_3$); 2.30-2.50 (8H, m, $4CH_2$); 5.58 (1H, s, CH); 7.11-7.13 (2H, d, CHO, ^3J = 6.8 Hz); 7.17-7.21 (1H, t, H_p, ^3J = 7.8 Hz); 7.27-7.31 (2H, dd, H_m, ^3J = 7.8 Hz, ^3J = 6.8 Hz).

3,3,6,6-tetramethyl-9-(4-methylphenyl)-1,8-dioxooctahydroxanthene **(3b)**: White powder, Yield (81%), mp 173-175 °C. FT-IR ($\bar{\upsilon}$, Cm^{-1}) (KBr disc): 3080 (CH_{arom}, Str.); 2950 (CH_{aliph}, Str.); 1670 (C=O Str.); 1540 (C=C Str.); 1380 (C-O Str.). ^{1}H NMR (400 MHz, $CDCl_3$) δ(ppm): 1.01 (6H, s, CH_3); 1.11 (6H, s, CH_3); 2.15 (4H, dd, CH_2); 2.26 (3H, s, CH_3); 2.47 (4H, s, CH_2); 4.8 (1H, s); 7.02 (2H, d, CH_{arom}, ^3J = 8.2 Hz); 7.19 (2H, d, CH_{arom}, ^3J = 8.2 Hz).

3,3,6,6-tetramethyl-9-(4-methoxyphenyl)-1,8-dioxooctahydroxanthene **(3c)**: Gray powder, Yield (82%), mp 238-239 °C. FT-IR ($\bar{\upsilon}$, Cm^{-1}) (KBr disc): 3080 (CH_{arom}, Str.); 3000 (CH_{aliph}, Str.); 1685 (C=O Str.); 1530 (C=C Str.); 1380 (C-O Str.). ^{1}H NMR (400 MHz, $CDCl_3$) δ(ppm): 1.01 (6H, s, CH_3); 1.11 (6H, s, CH_3); 2.22 (4H, dd, CH_2); 2.47 (4H, s, CH_2); 2.47 (1H, s, CH); 3.74 (3H, s, OCH_3); 6.7 (2H, d, CH_{arom}, ^3J = 8.4 Hz); 7.21 (2H, d, CH_{arom}, ^3J = 8.4 Hz).

Table 2
Synthesis of 1,8-dioxooctahydroxanthene derivatives in the presence of $Zr(HSO_4)_4$ and in optimal reaction conditions.

Entry	R	Product	Time (min)	Yield %	Mp (°C)	Mp (Lit)
1	C_6H_5	3a	40	86	201-203	205-206 (37)
2	*p*-$CH_3C_6H_4$	3b	50	81	229-230	222-225 (38)
3	*p*- $CH_3OC_6H_4$	3c	55	82	251-252	248-250 (37)
4	*m*- $CH_3OC_6H_4$	3d	35	92	304-305	308-310 (38)
5	*p*-Cl C_6H_4	3e	40	88	240-241	237-238 (37)
6	*p*-NO_2 C_6H_4	3f	35	94	230-032	226-228 (37)
7	*p*-OHC_6H_4	3g	60	80	254-255	250-251 (37)
8	2,4- di-Cl-C_6H_4	3h	30	95	239-241	247-248 (37)
9	*p*-$(Me)_2N$ C_6H_4	3i	60	72	295-297	-
10	*m*-Cl C_6H_4	3j	40	88	179-181	185-186 (39)

3,3,6,6-tetramethyl-9-(3-methoxyphenyl)-1,8-dioxooctahydroxanthene **(3d)**: White powder, Yield (92%), mp 159-160 °C. FT-IR ($\bar{\upsilon}$, Cm^{-1}) (KBr disc): 3100 (CH_{arom}, Str.); 3000 (CH_{aliph}, Str.); 1580 (C=O Str.); 1520 (C=C Str.); 1370 (C-O Str.). ^1H NMR (400 MHz, $CDCl_3$) δ(ppm): 1.12 (6H, s, CH_3); 1.25 (6H, s, CH_3); 2.30-2.48 (8H, m, CH_2); 3.75 (3H, s, OCH_3); 5.53 (1H, s, CH); 6.68-7.22 (4H, m, CH_{arom}).

Antibacterial activity

The antibacterial activity of the synthesized compounds was determined by agar disk diffusion method against gram-positive (S. aureus bacteria) and gram-negative (E. coli bacteria) microorganisms. For this purpose, Briefly, Mueller Hinton agar culture medium with a thickness of 5 mm was prepared first. Then a suspension of the desired bacteria was prepared with 0.5 *McFarland* turbidity standards. Next, 50 µl of fresh bacterial culture was pipetted in the center of sterile petri dish containing the prepared culture medium and dispersed well by sterile swap. Next, filter paper disks containing a concentration of 2000 µg/ml of the synthesized products (3a-3j) are placed on the surface of the agar previously inoculated with the desired bacterium. The plates were then incubated for 24 hours at 37°C. The inhibition diameters zones formed around each disk were measured by using a ruler. Each experiment was repeated three times and the average diameter of the growth inhibition zone was calculated.

Results and discussion

In order to optimize the reaction parameters, the catalytic activity of $Zr(HSO_4)_4$ in the synthesis of 1,8-dioxoctahydroxanthene derivatives under different reaction conditions was investigated. For this purpose, Reaction efficiency and reaction rate in different catalytic values and different solvents were investigated. Condensation reaction of benzaldehyde and dimedone was selected as a model reaction. As shown in Table 1, among the tested solvents such as water, THF, $CHCl_3$, CH_3CN, CH_3CH_2OH and a solvent-free system, the best result was obtained after 40 min under solvent-free conditions in good yield (86%) (Table 1, Entry 7). When the same reaction was performed in the absence of the catalyst, the corresponding product was obtained in trace (<10%) (Table 1, Entry 9). However, the presence of $Zr(HSO_4)_4$ in the amount of more than 30 mol%, the efficiency and reaction speed did not improve (Table 1, Entry 8)

After determining the optimal reaction conditions, aldol condensation reaction between dimedone and benzaldehyde derivatives was performed in the presence of $Zr(HSO_4)_4$ catalyst and in optimal reaction conditions (Table 2). All the products were characterized by ^1HNMR and IR spectra and compared with previous articles.

Table 3
Inhibition zones (mm) of synthesized 1,8-dioxooctahydroxanthenes derivatives against against gram-positive (S. aureus bacteria) and gram-negative (E. coli bacteria) microorganisms by the disc diffusion method.

Entry	Product	S. aureus	E. coli
1	3a	4*	0**
2	3b	3.5	0
3	3c	0	0
4	3d	0	0
5	3e	0	0
6	3f	0	0
7	3g	0	0
8	3h	15	7.5
9	3i	4	0
10	3j	3	0

* Numbers are reported in millimeters.
** The numbers reported are the inhibitions halos formation around the disk.

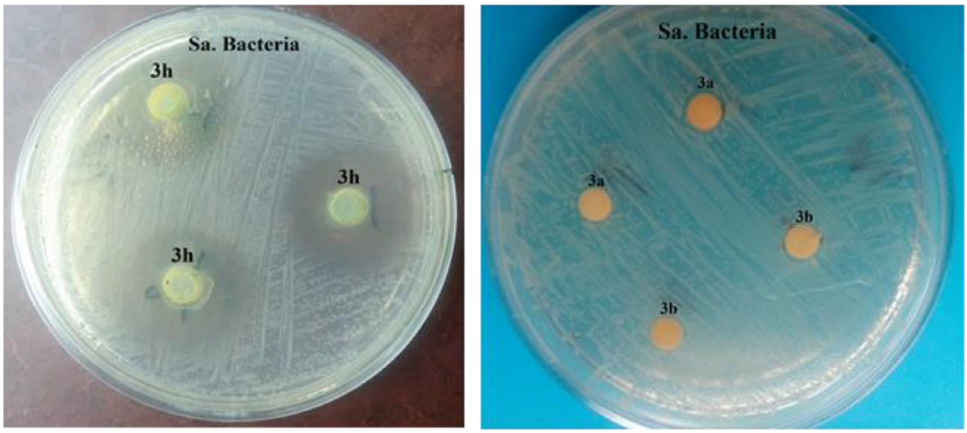


Fig. 1. Perform antibacterial test by disk diffusion method against gram-positive (S. aureus bacteria) and gram-negative (E. coli bacteria) microorganisms.

Antimicrobial activities of 1,8-dioxo-octahydroxanthene derivatives

The antibacterial properties of all synthesized compounds (3a-3j) were investigated by agar disk diffusion method against gram-positive (S. aureus bacteria) and gram-negative (E. coli bacteria) microorganisms. The results are reported in Table 3 and Fig. 1.

Recovery of catalyst

Recycling of heterogeneous catalysts after the end of the reaction and its reusability with the least change in the reaction efficiency can be considered one of the most important advantages of this type of catalyst. In order to investigate the recovery of $Zr(HSO_4)_4$ catalyst, after each use of this catalyst, we washed it with ethanol and water and dried it in the oven and then participated in the same reaction. The results show that the catalyst can be used up to three run without significantly changing the reaction efficiency (Fig. 2).

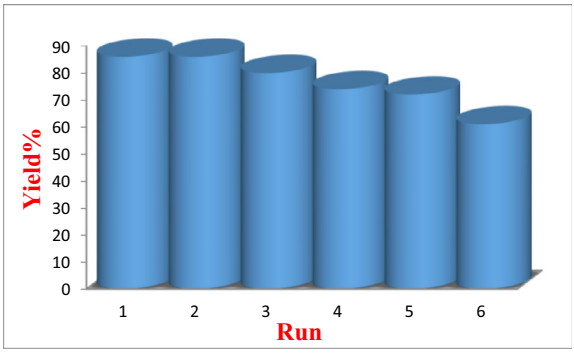
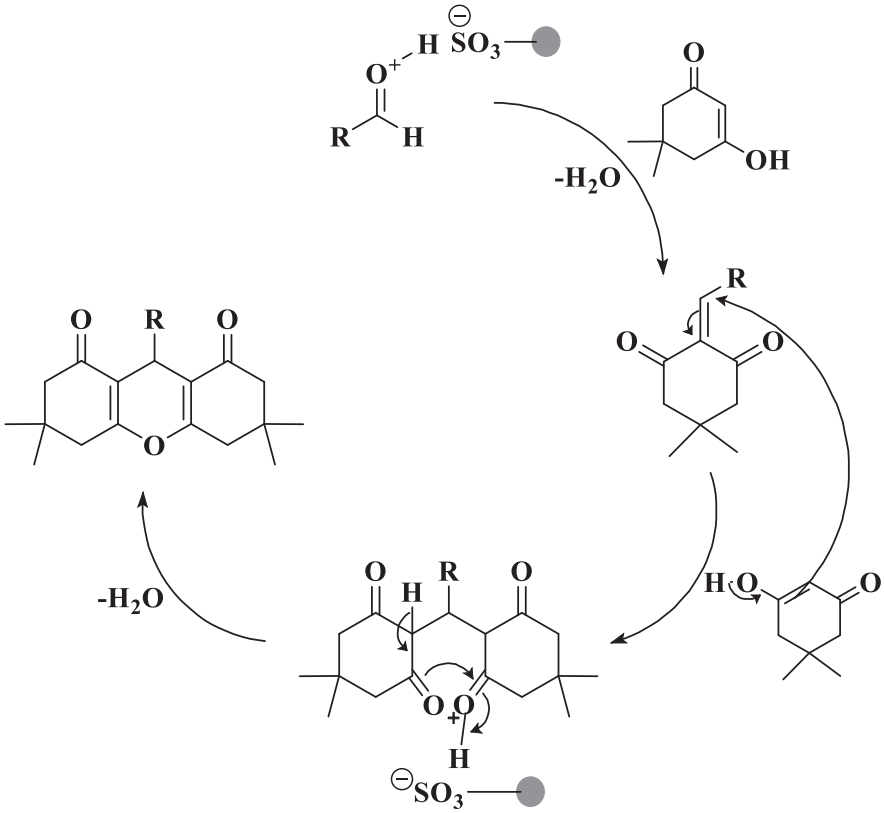


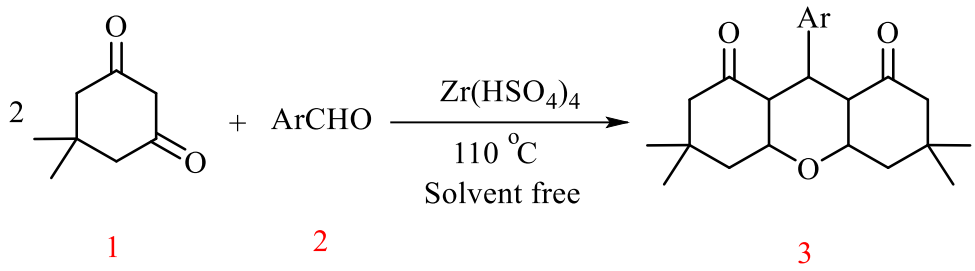
Fig. 2. The reusability of $Zr(HSO_4)_4$ catalyst for the preparation of 3,3,6,6-tetramethyl-9-phenyl-1,8-dioxoocta hydroxanthene (3a).



Scheme 2. Proposed mechanism for the synthesis of 1,8-dioxooctahydroxanthene in the presence of $Zr(HSO_4)_4$ as catalyst.

The proposed mechanism is shown in Scheme 2. At first, SO_3H groups, as an acidic agent, activates the carbonyl aldehyde group. Then dimedone attacks this intermediate. In the next step, by increasing Michael and the removal of a water molecule, the products are synthesized.

Table 4 shows a comparison between the methods reported in the articles and the method performed in this work.



Scheme 1. synthesis of 1,8-dioxooctahydroxanthenes.

Table 4
A comparison of the methods used in the articles and the method used in this work.

Entry	Catalyst	Conditions	Reaction time (min)	Yields%	Ref.
1	PEG	H_2O/reflux	~ 180	~ 90	[39]
2	activated zinc metal and solid NH_4C	Solvent free /MW	~ 50	~ 90	[40]
3	1-butyl-3-methylimidazoliumtetrafluoroborate (BMIF)	solvent free/~200 °C	~ 60	~ 90	[41]
4	Fe nanoparticles loaded in zeolite X (Fe-X)	Solvent-free, 90°C	~ 40	~ 90	[42]
5	$SO_3H@Fe_3O_4$ magnetic nanocatalyst	Solvent-free, 80 °C	~ 20	~ 90	[43]
6	$Zr(HSO_4)_4$	Solvent-free, 110°C	35-60	72-95	This work

Conclusion

In summary, in this study we introduced compound $Zr(HSO_4)_4$ as a green, effective and recyclable catalyst for the one pot synthesis of various 1,8-dioxooctahydroxanthenes derivatives. Some advantages of this protocol include a simple reaction set-up, high products yields and elimination of toxic solvents.

Declaration of Competing Interests

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data Availability

Data will be made available on request.

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.mex.2022.101832.

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