

Efficient and reusable Pumice supported perchloric acid catalyst for concise and effective synthesis of 1,8-dioxo-octahydroxanthenes under solvent free condition

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CHRONICLE

Article history:

Received March 20, 2023

Received in revised form

June 9, 2023

Accepted August 17, 2023

Available online

August 17, 2023

Keywords:

Heterogeneous Catalyst

Pumice@HClO₄

Dimedone

1,8-Dioxo-octahydroxanthene

Solvent Free Reaction

ABSTRACT

In the present investigation, a library of 1,8-dioxo-octahydroxanthene derivatives have been synthesised by the cyclo-condensation reaction of aryl aldehyde with two moles of dimedone under solvent free condition in the presence of Novel Pumice supported perchloric acid (Pumice@HClO₄) as a less expensive, recyclable, thermodynamically stable and non-toxic catalyst. The significant advantages of this novel protocol are experimental simplicity, easy work-up procedure, good to excellent yields of product and more sustainable as the efficiency of the readily available catalytic material.

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1. Introduction

Naturally occurring pumice composed of a high percentage of silica which is a good site to convert pumice into highly active supported catalytic materials.¹ The pumice supported catalytic materials like Pumice supported sulfonic acid,² Pumice supported perchloric acid,³ Pumice-modified cellulose fibre,⁴ and pumice based metal catalyst⁵ have been used to speed up many organic reactions. The advantages of the use of these catalytic materials are low cost, no toxicity, high porosity and heterogeneous nature which removes the obstacles in the scale up process of the reaction.⁶

In the last decades, 1,8-dioxo-octahydroxanthenes have much more interest due to its important applications in dyes industries, laser industries and also some derivative has been used for visualisation of bio-molecules with the view of its fluorescent properties.⁷⁻¹⁰ Xanthendione are found as an important core in various natural products. Xanthene and their derivatives have shown crucial antimicrobial,¹¹ antimycobacterial,¹² antimalarial,¹³ anticancer,¹⁴ antihypertensive,¹⁵ anti-inflammatory,¹⁶ antifungal,¹⁷ antiplatelet,¹⁸ antioxidant,¹⁹ etc. Some bioactive compounds containing xanthene nucleus are shown in **Fig. 1**.

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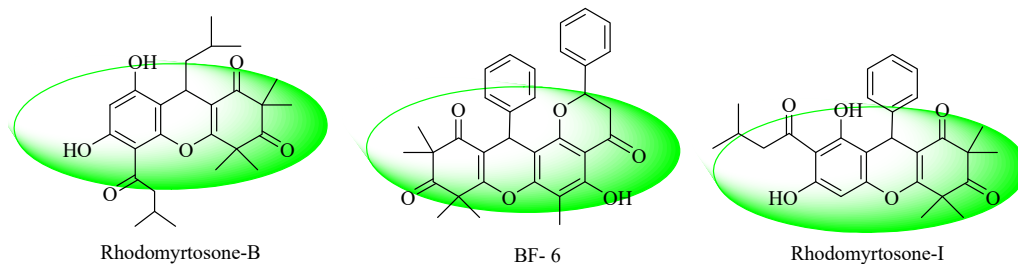
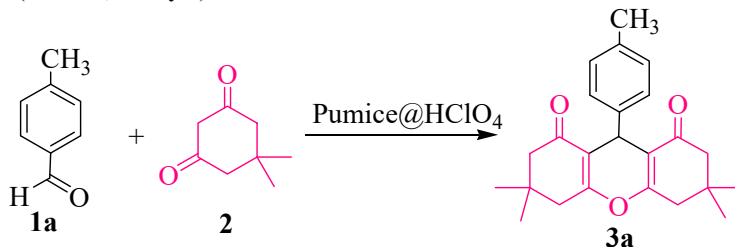


Fig. 1. Examples of bioactive 1,8-dioxooctahydroxanthene derivatives

Many reports are available in the literature for the synthesis of 1,8-dioxo-octahydroxanthene by the condensation reaction of active methylene compounds with aldehydes in the presence of different catalytic materials such as Fe₂O₃ NPs,²⁰ TMSCl,²¹ Perlite NPs@IL/ZrCl₄,²² β-cyclodextrin grafted with butyl sulfonic acid,²³ ZnS-Fe₂O₃-Ag Nanocomposite,²⁴ Barium Perchlorate,²⁵ TiO₂/[(SO₄)²⁻],²⁶ thiourea dioxide,²⁷ FeNP@SBA-15,²⁸ ZnO NPs,²⁹ KF/Al₂O₃ or Silica Sulfate,³⁰ [HBIM][BF₄]/US,³¹ Amberlyst-15,³² Cyanuric chloride,³³ Dowex-50W,³⁴ *p*-dodecyl benzene sulphonic acid,³⁵ SbCl₃/SiO₂,³⁶ silica sulphuric acid,³⁷ HClO₄-SiO₂,³⁸ and NaHSO₄-SiO₂,³⁹ triethylbenzyl ammonium chloride,⁴⁰ *p*-TSA,⁴¹ etc. Although, in continuation of our investigation in view of the development of new eco-friendly routes for the synthesis of bioactive compounds using a novel less toxic catalyst,^{2,3} we describe an effective and rapid method for the synthesis of 1,8-dioxo-octahydroxanthene using recyclable new pumice@HClO₄ as a catalyst. This study showed the synthesis of bioactive heterocyclic compounds that can be used in several applications, which reflects the high importance of applied organic chemistry in computational as well as different fields as reported before in a lot of scientific papers published before.⁴²⁻⁴⁶

2. Results and Discussion

To select the optimum condition, the reaction of dimedone (3 mmol) with 4-methyl benzaldehyde (1.5 mmol) was chosen as a model reaction (**Scheme 1**). Initially, the reaction mixture was ground in presence of pumice@HClO₄ catalyst for 30 min using mortar and pestle, but the result is no reaction. Then the reaction mixture was stirred in the presence of ethanol as a solvent for 3 hrs, which also gives negative results. After that the reaction mixture was heated in an oil bath at different temperatures such as 80, 100 and 120°C respectively, as shown in **Table 1**. However, the desired product obtained at 120°C with good yield (**Table 1, Entry 5**).



Scheme 1: Model reaction of Synthesis of 1,8-dioxo-octahydroxanthene derivative (**3a**).

Table 1. Optimization of condition for the synthesis of 1,8-dioxo-octahydroxanthene (**3a**).

Entry	Solvent system	Condition	Time (min)	Yield (%)
1	Solvent free	Grinding at RT	30	NR
2	Ethanol	Stirring at RT	180	NR
3	Solvent free	Heating at 80°C	180	Trace
4	Solvent free	Heating at 100°C	180	50
5	Solvent free	Heating at 120°C	30	85

4-methyl benzaldehyde (1.5 mmol), dimedone (3 mmol), pumice@HClO₄ (125 mg)

Next to find out the optimal quantity of the catalyst pumice@HClO₄, the model reaction was carried out under the solvent free conditions using variant quantities of catalyst at 120°C as shown in **Table 2**. The excellent result was obtained with the use of 125 mg of catalyst in a short time (**Table 2, Entry 3**). The use of excess quantities of the catalyst did not show remarkable increment in the yield.

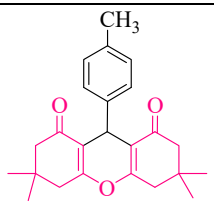
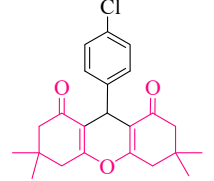
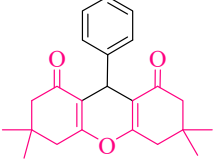
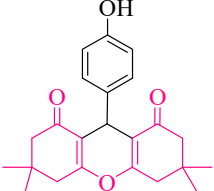
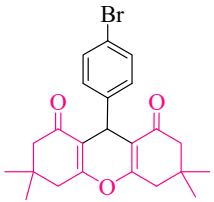
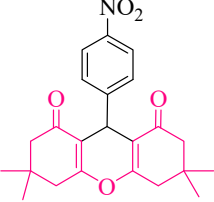
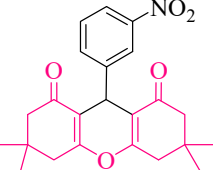
Table 2. Optimization of amount of catalyst for the synthesis of 1,8-dioxo-octahydroxanthene derivative (**3a**)

Entry	Amount of Catalyst (mg)	Time (min)	Yield (%)
1	75	30	60
2	100	30	75
3	125	30	85
4	150	30	86

4-methyl benzaldehyde (1.5 mmol), dimedone (3 mmol), Solvent Free heating at 120°C

To expand the scope of the protocol with the optimized condition, further reaction was carried out with differently substituted benzaldehyde having both electron-donating as well as withdrawing groups. But no noteworthy change in the yield and time of the reaction as summarized in **Table 3**. The heterogeneous pumice@HClO₄ catalyst was separated during purification of the targeted products by recrystallisation. During recrystallisation, it remained immiscible in the solvent hence easily separated by filtration. After separation, the obtained catalyst was washed by hot ethanol followed by ethyl acetate 2-3 times and can be reused for further reaction. The comparative study of the benefit of greener pumice@HClO₄ assisted protocol with the other reported method is shown in **Table 4**.

Table 3. Synthesis of 1,8-dioxo-octahydroxanthene derivatives (**3a-m**)

Entry	Xanthenedione derivative	Time (min.)	Yield (%)	M.P. (°C)	
				Found	Lit. Ref
3a		30	85	210-212	218-220 ²⁰
3b		30	82	229-231	230-232 ²⁰
3c		35	88	208-210	205-207 ²⁰
3d		35	80	246-248	245-247 ²⁰
3e		30	83	238-240	240-242 ²⁷
3f		40	78	218-220	218-220 ²⁰
3g		35	75	174-176	171-173 ²²

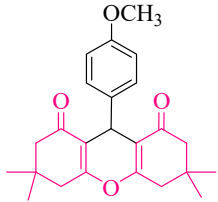
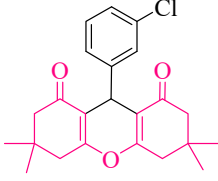
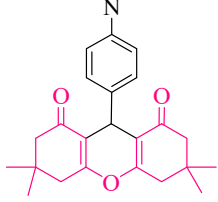
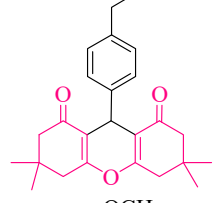
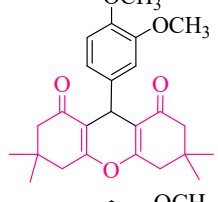
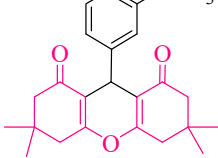
3h		30	82	244-246	240-242 ²⁰
3i		30	85	178-180	184-186 ²⁶
3j		35	82	218-220	221-222 ²⁶
3k		30	80	214-216	--
3l		35	76	186-188	--
3m		30	75	162-164	160-162 ²⁷

Table 4. Comparison of pumice@HClO₄ with other literature reported catalyst in the synthesis of 1,8-dioxo-octahydroxanthene

Sr. No.	Catalyst	Condition	Time (min)	Yield (%)	References
1	Fe ₂ O ₃ NPs	Stirred at RT in EtOH	120	90	20
2	TMSCl	Reflux in CH ₃ CN	90	95	21
3	perlite-NPs@IL/ZrCl ₄	Solvent free heating at 80°C	160	89	22
4	β-CD-BSA	Reflux in H ₂ O	45	90	23
5	[Hbim]BF ₄	US-MeOH at RT	60	75	31
6	pumice@HClO ₄	Solvent free heating at 120°C	30	85	Present work

3. Conclusions

We have developed a rapid, efficient, and convenient protocol for the synthesis of 1,8-dioxo-octahydroxanthenes by

condensation reaction of aryl aldehydes and dimedone under solvent free condition in presence of pumice@HClO₄ as a less expensive, recyclable, thermodynamically stable and non-toxic catalyst. The products can be easily isolated by simple work up procedures and purified by recrystallisation without use of column chromatography technique. The significant advantages of the present protocol are experimental simplicity, solvent free reaction condition, short reaction time, good to excellent yields of product, and more sustainable as the efficiency of the readily available catalytic material. These reports proposed a novel, useful and attractive route for the synthesis of 1,8-dioxo-octahydroxanthenes.

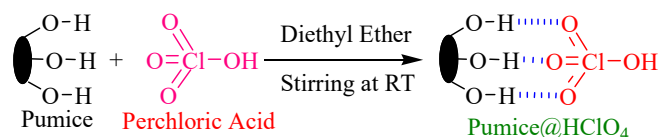
Acknowledgements

The authors are grateful to the Principal of Arts, Science and Commerce College, Rahata for providing all necessary facilities and constant encouragement. We are also thankful to SAIF, Punjab University, Chandigarh for providing spectral analysis.

4. Experimental

4.1. Materials and Methods

Melting points were recorded in an open capillary and are uncorrected. IR spectra were recorded on a Perkin-Elmer FTIR spectrophotometer. The ¹H-NMR and ¹³C-NMR spectra were recorded on a BRUCKER AVANCE NEO 500 MHz NMR spectrometer in CDCl₃ using TMS as an internal standard. Mass spectra were recorded on a MALDI SYNAPT XS HD Mass spectrometer. TLC was carried out by Al-plates pre-coated with silica gel to check the purity of the compounds. The pumice@HClO₄ has been prepared by the previously reported method³ as shown in **Scheme 2**.

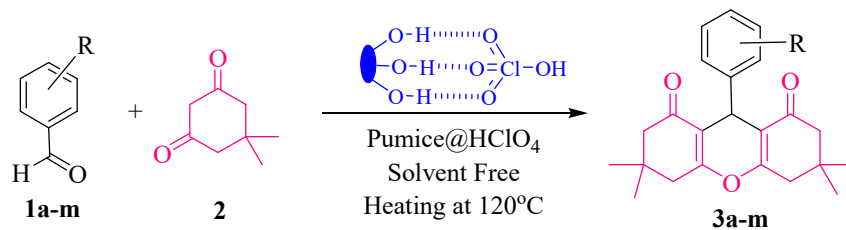


Scheme 2: Preparation of Pumice@HClO₄

4.2. General procedure

General procedure for the synthesis of 1,8-dioxo-octahydroxanthene derivatives (3a-m).

The mixture of aryl aldehyde (1.5 mmol), 1,3-cyclohexadione (3 mmol) and pumice@HClO₄ (125 mg) was taken in a round bottom flask in the absence of solvent (**Scheme 3**). The resulting reaction mixture was subjected for heating in an oil bath at 120°C for appropriate time until all the aldehyde is consumed. The progress of the reaction was confirmed by TLC in n-hexane:ethyl acetate (9:1). After completion of the reaction, the obtained solid was filtered off. The obtained solid was purified by recrystallisation technique using ethanol. During the purification process, the catalyst pumice@HClO₄ remains immiscible in ethanol hence after the filtration at hot condition it remains on filter paper and thus separated from the reaction mixture. Then after cooling the mother liquor the product was separate out which was filtered off and dried.



Scheme 3: Synthesis of 1,8-dioxo-octahydroxanthenes (**3a-m**).

4.3 Physical and Spectral Data

3,4,6,7-tetrahydro-3,3,6,6-tetramethyl-9-p-tolyl-2H-xanthene-1,8(5H,9H)-dione (3a):

Yield 85%; m.p. 210-212°C; FT-IR (ν in cm⁻¹): 2961.36 (–CH), 1658.21 (–C=O), 1509.46 (C=C), 1217.10 (–C–O); ¹H NMR (500 MHz, CDCl₃, ppm): δ 0.97 (s, 6H, –CH₃ X 2), 1.09 (s, 6H, –CH₃ X 2), 2.17-2.21 (m, 4H, –CH₂ X 2), 2.24 (s, 3H, Ar-CH₃), 2.45 (s, 4H, –CH₂ X 2), 4.71 (s, 1H, –CH), 7.01 (d, *J* = 8 Hz, 2H, Ar-H), 7.16 (d, *J* = 8 Hz, 2H, Ar-H).

3,4,6,7-tetrahydro-3,3,6,6-tetramethyl-9-phenyl-2H-xanthene-1,8(5H,9H)-dione (3c):

Yield 88%; m.p. 208-210°C; ¹H NMR (500 MHz, CDCl₃, ppm): δ 0.99 (s, 6H, -CH₃ X 2), 1.09 (s, 6H, -CH₃ X 2), 2.15-2.24 (m, 4H, -CH₂ X 2), 2.46 (s, 4H, -CH₂ X 2), 4.75 (s, 1H, -CH), 7.09 (t, *J* = 7.3 Hz, 1H, Ar-H), 7.21 (t, *J* = 7.5 Hz, 2H, Ar-H), 7.28 (t, *J* = 7.3 Hz, 2H, Ar-H).

3,4,6,7-tetrahydro-9-(4-hydroxyphenyl)-3,3,6,6-tetramethyl-2H-xanthene-1,8(5H,9H)-dione (3d):

Yield 80%; m.p. 246-248°C; FT-IR (ν in cm⁻¹): 3413.33 (-OH), 2962.50 (C-H), 1638.65 (C=O), 1512.87 (C=C), 1202.33 (C-O); ¹H NMR (500 MHz, CDCl₃, ppm): δ 0.99 (s, 6H, -CH₃ X 2), 1.09 (s, 6H, -CH₃ X 2), 2.17-2.25 (m, 4H, -CH₂ X 2), 2.46 (s, 4H, -CH₂ X 2), 4.66 (s, 1H, -CH), 6.54 (d, *J* = 8.5 Hz, 2H, Ar-H), 7.06 (d, *J* = 8.5 Hz, 2H, Ar-H); ¹³C NMR (100 MHz, CDCl₃, ppm): δ 27.41, 29.16, 30.96, 32.37, 40.86, 50.77, 115.27, 115.90, 129.34, 135.55, 154.74, 162.45, 197.26.

3,4,6,7-tetrahydro-3,3,6,6-tetramethyl-9-(4-nitrophenyl)-2H-xanthene-1,8(5H,9H)-dione (3f):

Yield 78%; m.p. 218-220°C; FT-IR (ν in cm⁻¹): 2960.30 (C-H), 1637.96 (C=O), 1515.17 (C=C), 1202.26 (C-O); ¹H NMR (500 MHz, CDCl₃, ppm): δ 0.99 (s, 6H, -CH₃ X 2), 1.12 (s, 6H, -CH₃ X 2), 2.15-2.27 (m, 4H, -CH₂ X 2), 2.49 (s, 4H, -CH₂ X 2), 4.82 (s, 1H, -CH), 7.47 (d, *J* = 8.7 Hz, 2H, Ar-H), 8.09 (d, *J* = 8.7 Hz, 2H, Ar-H); Mass (m+1) mode m/z = 396.1875.

9-(3-chlorophenyl)-3,4,6,7-tetrahydro-3,3,6,6-tetramethyl-2H-xanthene-1,8(5H,9H)-dione (3i):

Yield 85%; m.p. 178-180°C; ¹H NMR (500 MHz, CDCl₃, ppm): δ 1.01 (s, 6H, -CH₃ X 2), 1.10 (s, 6H, -CH₃ X 2), 2.17-2.26 (m, 4H, -CH₂ X 2), 2.48 (s, 4H, -CH₂ X 2), 4.73 (s, 1H, -CH), 7.08 (m, *J* = 1.1 Hz, 1H, Ar-H), 7.14 (m, *J* = 7.85 Hz, 1H, Ar-H), 7.22 (m, *J* = 1.5 Hz, 1H, Ar-H), 7.24 (m, *J* = 6.7 Hz, 1H, Ar-H); ¹³C NMR (100 MHz, CDCl₃, ppm): δ 27.39, 29.20, 31.74, 32.23, 40.86, 50.70, 115.12, 126.67, 127.04, 128.28, 129.24, 133.89, 146.09, 162.55, 196.29.

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