

An expedient and straightforward solvent-free synthesis of 1,8-dioxooctahydroxanthenes using eco-friendly aluminized polyborate catalyst

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Abstract

The present work deals with a catalytic application of aluminized polyborate as a mild solid acid catalyst for multicomponent synthesis of 1,8-dioxo-octahydroxanthenes under solvent-free condition at 100-110°C. It involves selective domino Knoevenagel-Michael reaction of one mole of aromatic aldehyde with two moles dimedone. The aluminized polyborate is simply synthesized by using boric acid and aluminium trichloride and characterized by FT-IR, XRD, SEM and EDAX techniques. The mild Lewis acidity, crystalline solid nature, stability and recyclability are significant features of the catalyst. The solvent-free condition, clean reaction profile, inexpensive and non-toxic catalyst, operational simplicity, good to excellent product yields, reduced reaction time and applicability to wide range of substrate are crucial features of this protocol.

Keywords: 1,8-dioxo-octahydroxanthene, aluminized polyborate, solvent-free condition, green protocol.

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1 | INTRODUCTION

Xanthene is annulated pyran heterocyclic compounds found as an important core of natural products, compounds of biological importance and also in fluorescent dyes.^[1] Due to presence of pyran framework they are associated with biological functions such as antiviral,^[2] anticancer,^[3] antioxidant,^[4] antimicrobial,^[5] analgesic,^[6] anti-inflammatory^[7] and antimalarial.^[8] Xanthene is privileged scaffold found in compounds having therapeutic importance (Figure 1).



allosteric modulators of the d-opioid receptor

osteogenic agents

estrogen receptor modulators

FIGURE 1 Structure of some biologically important xanthene nucleus

Beside this, due to their high molecular extinction coefficient as well as high quantum yield they also find application in dyes. They are widely used as pH-sensitive substance for the visualization of biomolecules and dyes.^[9] The fluorescent dyes such as fluorescein and rhodamine are utilized as a synthetic electrostatic chemosensors and super resolutions bioimaging of different biological samples.^[10] They are also extensively used as photodynamic sensitizers^[11] and also acts as corrosion inhibitors.^[12]

Nowadays, there is tremendous demand for synthetic approaches which reduces use of toxic material and produce products which find application for well being of society. It is a key challenge for chemist to follow the green chemistry principles for development of environmentally sustainable process.^[13] In this context, the multicomponent reactions (MCRs) have emerged as powerful tool for investigation of novel methodologies which involves three or more substrate components.^[14] The MCRs have significant contribution in synthesis of multi-functionalized complex organic compounds in single step with faster reaction rate and excellent yield.^[15] Along with this, solvent-free condition or use of green solvent, use of non-toxic reagents, reduce waste, clean and simple reaction profile and good atom economy are additional advantages of MCR approach.^[16] It is known that solvent itself is a key basis of waste, thus solvent-free reaction becomes one of the important way to minimize waste and make the process more greener.^[17]

Boric acid has made its place in development of newer catalytic system due to its ready availability, cheap and non-toxic nature.^[18] But the reactions in which temperature is above 100°C, boric acid converts into polymeric borate through elimination of water molecule. This water molecule may hamper progress of some reactions.^[19] This limitation gave insights for development of polymeric boric acid catalyst such as sulfated and aluminized polyborate with enhanced catalytic activity.^[20] The presence of aluminium and boron in aluminized polyborate generates mild Lewis acidic character and makes it suitable for catalytic applications.^[21]

In general, the straightforward synthesis of 1,8-dioxooctahydroxanthene involves onepot domino Knoevenagel-Michael reaction of two moles of dimedone with one mole of aromatic aldehydes. In literature several catalytic systems are reported for their synthesis such as [DDPA][HSO₄],^[22] Ni NPs@*N*-doped titania,^[23] C/TiO₂-SO₃H,^[24] ZnO,^[25] sulfated zirconia,^[26] Fe₃O₄@SiO₂ Imid-PMA,^[27] β-cyclodextrin,^[28] Bronsted acidic ionic liquids,^[29] Fe–Cr–Ni alloy nano-belts,^[30] ZnO-NPs,^[31] alumina-sulfuric acid,^[32] MSrGO NCs,^[33] Choline chloride,^[34] SmCl₃,^[35] PDNES,^[36] PVPP-BF₃,^[37] PSA,^[38] natural phosphate,^[39] n-TSA,^[40] Fe₂(SO₄)₃.7H₂O,^[41] barium perchlorate,^[42] Ag@CDNS-N/PMelamine,^[43] sawdust sulphonic acid,^[44] nano-WO₃-supported sulfonic acid,^[45] CoNP@SBA-15,^[46] PFPA,^[47] MnFe₂O₄,^[48] Fe₃O₄@PS@His[HSO₄⁻],^[49] FSM-16/AEPC-SO₃H ^[50] and zeolite nanoparticles.^[51] Accepted Article

All these reported methods have some beneficial features but at the same time lacks with one or more vital synthetic parameters such as use of costly and toxic catalyst, lengthy work-up, extended reaction time, low yield and use of non-green solvents. To overcome such problems there is still demand to develop novel methodologies with greener conditions. The biological importance of xanthene, need of newer environmental benign protocol and in continuation to our work^[52-54] herein, we report aluminized polyborate as mild solid acid catalysed expedient and efficient synthesis of 1,8-dioxo-octahydroxanthenes using two moles of dimedone and one mole of aromatic aldehyde under solvent-free condition at 100-110°C with excellent product yield in shorter time (Scheme 1).



SCHEME 1 General scheme for the synthesis of 1, 8-dioxo-octahydroxanthenes

2 | RESULTS AND DISCUSSION

2.1 | Synthesis of aluminized polyborate

Boric acid and aluminium chloride were used for the synthesis of aluminized polyborate catalyst following the procedure reported in the literature (Figure 2).^[21] The catalyst obtained is solid crystalline in nature having good stability with mild Lewis acidity. The fresh as well as recovered catalyst has been characterized by FT-IR, XRD, SEM and EDAX techniques. The results obtained specify formation of aluminized polyborate. The characterization data is provided in supplementary material associated with this article.



FIGURE 2 Outline for synthesis of aluminized polyborate

2.2 | Application of aluminized polyborate

The successful synthesis and characterization of aluminized polyborate motivated us to explore its catalytic function for the synthesis of 1,8-dioxo-octahydroxanthenes. Initially we started our study with choosing a reaction of one mole of benzaldehyde (**1a**) and two mole of

dimedone (2) as a model reaction for optimizing influence of solvent, temperature and amount of catalyst for the synthesis of 1,8-dioxo-octahydroxanthenes (Scheme 2).



SCHEME 2 Model reaction for synthesis of 1,8-dioxo-octahydroxanthenes (3a)

In primary study, we performed series of experiments on model reaction with aluminized polyborate catalyst in various solvent like EtOH, H_2O , MeOH, THF, DMF and solvent-free at different temperatures (Table 1). The reaction carried out at room temperature in various systems does not forward the reaction. This shows that heat energy is essential to carry the reaction. The outcome indicates, solvent-free reaction at 100-110°C is the most appropriate condition for synthesis of 1,8-dioxo-octahydroxanthene (Table 1, entry 13,14) in terms of clean reaction, excellent yield in short time.

Entry	Solvent	Catalyst, mg	Temperature, °C	Time, min	Yield, ^b %
1	H ₂ O	75	r.t.	30	
2	H_2O	75	60	30	25
3	H ₂ O	75	reflux	30	59
4	EtOH	75	r.t.	30	
5	EtOH	75	reflux	30	84
6	H ₂ O:EtOH	75	r.t.	30	
7	H ₂ O:EtOH	75	reflux	30	68
8	MeOH	75	reflux	30	66
9	THF	75	reflux	30	55
10	DMF	75	80	30	62
11	solvent-free	75	r.t.	30	
12	solvent-free	75	80	25	75
13	solvent-free	75	100	15	93
14	solvent-free	75	110	15	93

TABLE 1 Optimization of effect of solvent and temperature for synthesis of $(3a)^{a}$

^aReaction conditions: benzaldehyde (1 mmol), dimedone (2 mmol) and aluminized polyborate (75 mg). ^bIsolated yield.

With the model reaction, our next aim was to investigate the most suited amount of catalyst for conversion into product. The results obtained shows that in absence of catalyst there was no formation of product while with increase in amount of catalyst from 15 mg, 25 mg, 50 mg to 75 mg there is significant increase in product yields (Table 2). The 75 mg of aluminized polyborate is the optimistic amount of catalyst required for prompt synthesis of **3a** in excellent yield (Table 2, entry 5).

Entry	Catalyst, mg	Temperature	Time, min	Yield, ^b %
1	catalyst-free	100-110°C	60	trace
2	15	100-110°C	60	34
3	25	100-110°C	40	53
4	50	100-110°C	25	69
5	75	100-110°C	15	93
6	100	100-110°C	15	93

TABLE 2 Optimization of amount of catalyst for synthesis of $(3a)^a$

^aReaction conditions: benzaldehyde (1 mmol), dimedone (2 mmol) and aluminized polyborate. ^bIsolated yield.

It is important to state that solvent-free reaction at $100-110^{\circ}$ C with 75 mg aluminized polyborate is the optimum condition for synthesis of **3a**. With this optimized results, we further thought to extend our study to ensure scope, versatility and selectivity of this MCR for synthesis of 1,8-dioxo-octahydroxanthene derivatives using aromatic aldehydes having electron donar as well as electron withdrawing groups like -Me, -OMe, -Cl, -Br, -F, -OH and -NO₂ (Table 3). It is noteworthy to mention that reactions with all substrates resulted with good to excellent yields in short time. The sterically hindered substrates as well as heteroaromatic aldehyde also gave product with good yields.





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^aReaction conditions: aromatic aldehydes (1 mmol), dimedone (2 mmol) and aluminized polyborate (75 mg) at 100-110°C under solvent-free condition. ^bIsolated yield.

The acidic nature of aluminized polyborate catalyst was studied by potentiometric titration.^[21] An exactly weighed aluminized polyborate (0.1 g) was dissolved in water and glycerin (2:1) mixture and titrated against standard 0.1 N NaOH solution. The graph of titration data $\Delta mV/\Delta V$ was plotted against the volume of NaOH added. The Figure 3 shows that, the H⁺ ion concentration was found to be 19 mmol/g at equivalence point which supports the acidic character of aluminized polyborate catalyst.



FIGURE 3 Potentiometric titration curve of aluminized polyborate

In further study we explored the reusability of aluminized polyborate catalyst for synthesis of diverse 1,8-dioxo-octahydroxanthene derivatives. The catalyst recovered (mention in general procedure) was reused over four cycles in model reaction **3a** under optimized condition. It was found that there was no significant loss in catalytic activity in terms of product yield and reaction time (Figure 4). The recovered catalyst was characterized by FT-IR, XRD, SEM and EDAX techniques which indicates that there are no major changes in catalyst composition as compared with fresh catalyst.



FIGURE 4 Recyclability of aluminized polyborate catalyst

To evaluate the merits of this protocol, we compared with literature reported methods for the synthesis of 1,8-dioxo-octahydroxanthenes (Table 4). The results illustrate that, faster reaction rate, excellent product yields, low-cost catalyst and solvent-free conditions are some values of this method.

TABLE 4 The comparison of various literature reported methods with present protocol for the synthesis of 1, 8-dioxo-octahydroxanthenes

Entry	Catalyst and solvent	Catalyst amount	Temperatu re	Time	Yield (%)	Reference
1	[DDPA][HSO ₄], solvent-free	0.5 mmol	100°C	1-2 h	87-97	22
2	C/TiO_2 -SO ₃ H, solvent-free	0.2 g	120°C	0.5-2.5 h	85-94	24
3	sulfated zirconia, EtOH	15 wt%	70-80°C	8-12 h	84-95	26
4	Bronsted acidic ionic liquids, solvent-free	10 mol%	95-100°C	30-45 min	85-95	29
5	MSrGO-NCs, solvent-free	15 mg	80°C	0.8-3 h	80-97	33
6	PDNES, solvent-free	5 mol%	90°C	30-120 min	80-97	36
7	PSA, solvent-free	0.05 g	80°C	40-110 min	75-89	38
8	natural phosphate, EtOH	1.5 g	reflux	6.5-8.5 h	84-95	39
9	n-TSA, solvent-free	0.013 g	90°C	63-90 min	83-96	40
10	Fe ₂ (SO ₄) ₃ .7H ₂ O, solvent-free	10 mol%	120°C	1.0-4.5 h	71-94	41
11	CoNP@SBA-15, H ₂ O	0.1 mol%	60°C	0.5-1.5 h	88-98	46
12	FSM-16/AEPC-SO ₃ H, solvent-free	0.45 mol%	120°C	15-95 min	64-89	50
13	aluminized polyborate, solvent-free	75 mg	100- 110°C	14-25 min	85-94	Present work

3 | EXPERIMENTAL

3.1 | Materials and methods

The chemicals used in this study were of analytical grade and used without further purification. The progress of reaction was checked by TLC using silica G60 F254 plates (Merck). The melting points were measured in open capillary tube and are uncorrected. FT-

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IR spectra were obtained on Perkin-Elmer, 400 FT-IR spectrometer. XRD analysis is performed on Panalyticals X Pert Pro, X-Ray Diffractometer. The SEM images were recorded on JSM6100, (Jeol) Scanning Electron Microscope. The ¹H-NMR spectra were recorded on Bruker Avance 500MHz NMR spectrometer using CDCl₃ as solvent and TMS as internal standard. Mass spectra were recorded on Waters Q-ToF Micromass spectrometer.

3.2 | General procedure for synthesis of 1, 8-dioxo-octahydroxanthenes (3a-o)

A mixture of substituted aromatic aldehyde (**1a-o**, 1 mmol) and dimedone (**2**, 2 mmol) were heated under solvent-free condition at 100-110°C in presence of 75 mg of aluminized polyborate catalyst (for time, Table 3). The progress and completion of reaction was checked by thin layer chromatography using n-hexane and ethyl acetate (7:3) as mobile phase. After completion of reaction, 5 mL of ethanol was added and resulting mixture was warmed. The hot solution was filtered to remove any insoluble material. The pure solid products were obtained from mother liquor by filtration at room temperature. The filtrate was evaporated to recover the solid catalyst. This recovered catalyst was dried at 100°C for one hour and used for next cycle. The physical as well as IR, ¹H-NMR and mass spectral data are in good agreement with that reported in literature.

3.3 | Spectral data of some representative 1, 8-dioxooctahydroxanthenes

3,3,6,6-tetramethyl-9-phenyl-3,4,5,6,7,9-hexahydro-1H-xanthene-1,8-(2H)-dione (3a)

White solid, m.p. 204-206°C; IR (KBr): 3083, 3032, 2915, 1662, 1491, 1235, 841 cm⁻¹; ¹H-NMR (500 MHz, CDCl₃) δ: 0.98 (s, 6H, 2 x CH₃), 1.09 (s, 6H, 2 x CH₃), 2.14-2.24 (m, 4H, 2 x -CH₂), 2.46 (s, 4H, 2 x -CH₂), 4.74 (s, 1H, -CH), 7.08-7.29 (m, 5H, Ar-H) ppm; MS m/z = 351 (M+1).

9-(3,4-dimethoxyphenyl)-3,3,6,6-tetramethyl-3,4,5,6,7,9-hexahydro-1H-xanthene-1,8-(2H)-dione (3g) Accepted Article

White solid, m.p. 182-184°C; IR (KBr): 3083, 3005, 2955, 1667, 1624, 1466, 1263, 1139 cm⁻¹; ¹H-NMR (500 MHz, CDCl₃) δ: 1.00 (s, 6H, 2 x CH₃), 1.10 (s, 6H, 2 x CH₃), 2.17-2.25 (m, 4H, 2 x –CH₂), 2.45 (s, 4H, 2 x –CH₂), 3.79 (s, 3H, OCH₃), 3.85 (s, 3H, OCH₃), 4.70 (s, 1H, -CH), 6.70 (d, 1H, J=8 Hz, Ar-H), 6.75 (dd, 1H, J=8 Hz & 2 Hz, Ar-H), 6.90 (d, 1H, J=2 Hz, Ar-H); MS m/z = 412 (M+1)..

9-(4-nitrophenyl)-3, 3, 6, 6-tetramethyl-3, 4, 5, 6, 7, 9-hexahydro-1H-xanthene-1, 8-(2H)dione (3i)

White solid, m.p. 228-229°C; IR (KBr): 3082, 3039, 2960, 1659, 1620, 1468, 1361, 1234, 866 cm⁻¹; ¹H-NMR (500 MHz, CDCl₃) δ: 0.99 (s, 6H, 2 x CH₃), 1.12 (s, 6H, 2 x CH₃), 2.15-2.27 (m, 4H, 2 x –CH₂), 2.49 (s, 4H, 2 x –CH₂), 4.82 (s, 1H, -CH), 7.46 (d, 2H, J=7.5 Hz, Ar-H), 8.08 (d, 2H, J=7.5 Hz, Ar-H) ppm; MS m/z = 396 (M+1).

9-(4-bromophenyl)-3,3,6,6-tetramethyl-3,4,5,6,7,9-hexahydro-1H-xanthene-1,8-(2H)dione (3j)

White solid, m.p. 240-241°C; IR (KBr): 3080, 3026, 2953, 1660, 1625, 1468, 1260, 1003, 849 cm⁻¹; ¹H-NMR (500 MHz, CDCl₃) δ: 0.98 (s, 6H, 2 x CH₃), 1.10 (s, 6H, 2 x CH₃), 2.14-2.25 (m, 4H, 2 x –CH₂), 2.42 (s, 4H, 2 x –CH₂), 4.69 (s, 1H, -CH), 7.15 (d, 2H, J=9 Hz, Ar-H), 7.32 (d, 2H, J=9 Hz, Ar-H) ppm; MS m/z = 430 (M+1).

9-(4-chlorophenyl)-3,3,6,6-tetramethyl-3,4,5,6,7,9-hexahydro-1H-xanthene-1,8-(2H)dione (3m)

White solid, m.p. 231-233°C; IR (KBr): 3090, 3030, 2962, 1678, 1625, 1469, 1361, 849 cm⁻¹; ¹H-NMR (500 MHz, CDCl₃) δ: 0.98 (s, 6H, 2 x CH₃), 1.10 (s, 6H, 2 x CH₃), 2.14-2.25 (m, 4H, 2 x –CH₂), 2.46 (s, 4H, 2 x –CH₂), 4.70 (s, 1H, -CH), 7.17 (d, 2H, J=8.7 Hz, Ar-H), 7.21 (d, 2H, J=8.7 Hz, Ar-H) ppm; MS m/z = 386 (M+1).

9-(p-tolylphenyl)-3, 3, 6, 6-tetramethyl-3, 4, 5, 6, 7, 9-hexahydro-1H-xanthene-1, 8-(2H)dione (3n) White solid, m.p. 213-215°C; IR (KBr): 3039, 3015, 2932, 1678, 1623, 1466, 1296 cm⁻¹; ¹H-NMR (500 MHz, CDCl₃) δ: 0.99 (s, 6H, 2 x CH₃), 1.09 (s, 6H, 2 x CH₃), 2.14-2.21 (m, 4H, 2 x –CH₂), 2.23 (s, 3H), 2.45 (s, 4H, 2 x –CH₂), 4.70 (s, 1H, -CH), 7.00 (d, 2H, J=8 Hz, Ar-H), 7.16 (d, 2H, J=8 Hz, Ar-H); MS m/z = 365 (M+1).

3.5 | Reaction mechanism

The plausible mechanism for the synthesis of 1,8-dioxo-octahydroxanthenes is depicted as follows (Scheme 3). The aluminized polyborate activates the carbonyl group of dimedone as well as aldehyde due to its Lewis acid character. Initially there is Knoevenagel condensation reaction between aldehyde and first dimedone molecule to form Michael acceptor intermediate. The activated intermediate undergoes Michael addition reaction with second molecule of dimedone which undergoes cyclization with loss of water molecule to yield 1,8-dioxo-octahydroxanthenes.



aluminized polyborate

SCHEME 3 Plausible mechanism for synthesis of 1,8-dioxo-octahydroxanthenes.

4 | CONCLUSION

In this work we have developed newer expedient, green and straightforward protocol for the one-pot domino Knoevenagel-Michael synthesis of 1,8-dioxo-octahydroxanthenes prompted by aluminized polyborate as a mild solid Lewis acid catalyst. The catalyst is characterized by FT-IR, XRD, SEM and EDAX analyses. The catalyst is non-toxic and inexpensive with good catalytic activity. The significant features of this protocol are faster reaction, solvent-free condition, excellent yield, reusable catalyst, no side reactions, easy work-up and applicable to wide range of substrates. This cost-effective and eco-sustainable methodology will be a good contribution to green chemistry.

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SUPPORTING INFORMATION

Additional information associated with this article may be found online in the supporting information at the end of this article.

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