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Original scientific paper

Facile and rapid synthesis of diverse xanthene derivatives using lanthanum(III) chloride/chloroacetic acid as an efficient and reusable catalytic system under solvent-free conditions

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Abstract: $\text{LaCl}_3/\text{ClCH}_2\text{COOH}$ was used as an efficient, and recyclable catalytic system for synthesis of 11*H*-benzo[*a*]xanthene-11-one, hexahydro-1*H*-xanthene-1,8(2*H*)-dione and 11-aryl-10*H*-diindeno[1,2-*b*:2',1'-*e*]pyran-10,12(1*H*)-dione derivatives *via* a one-pot three-component reaction of aldehydes, 2-naphthol, and cyclic 1,3-dicarbonyl compounds. The reactions proceeded rapidly at 70 °C under solvent-free conditions and the desired products were obtained in good to excellent yields.

Keywords: multi-component reactions (MCRs); 11*H*-benzo[*a*]xanthene; hexahydro-1*H*-xanthene, 1,3-dicarbonyl compounds; solvent-free conditions; green chemistry.

INTRODUCTION

In the past few decades, heterocyclic chemistry has become one of the most important disciplines in organic synthesis and pharmaceutical chemistry.¹ A large numbers of the synthesized compounds have emerged as active pharmaceutical components in several drugs due to their potential anti-inflammatory,^{2,3} anti-tumor,⁴ anti-hyperlipidemic,⁵ anti-hypertensive,⁶ anti-HIV infections⁷ and several other biological properties.^{8,9}

Xanthenes are an important class of heterocyclic compounds with remarkable biological and medicinal properties, such as antiviral, antibacterial and anti-inflammatory activity.^{10–13} Furthermore, they are used as leuco-dyes, pH sensitive fluorescent materials, and in laser technologies.^{14–16} Among this class of molecules, xanthone is a prominent structural motif found in numerous natural products and synthetic compounds with important biological activities.^{17–21}

Multi-component reactions (MCRs) have emerged as efficient and powerful tools in organic and medicinal chemistry due to their ability to synthesize organic

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molecules with higher efficiency and atom economy in a single step from three or more reactants. Moreover, MCRs offer the advantage of simplicity and synthetic efficiency over conventional chemical reactions.²² Therefore, developing new MCRs and improving known MCRs for the synthesis of diverse groups of compounds, especially ones that are biologically active, have gained great attention in current organic synthesis.^{23–25} In addition, solvent-free conditions make synthesis simpler, save energy, and prevent solvent waste, hazards, and toxicity.^{26–28} It therefore remains a challenge to develop multi-component reactions with suitable heterogeneous catalysts.

An interesting example of MCRs is the synthesis of xanthene derivatives that can be realized by condensation of aldehydes with β -naphthol, cyclic 1,3-dicarbonyl compounds and/or a mixture of aldehyde and cyclic 1,3-dicarbonyl compounds. Various catalysts, such as $\text{KAl}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$,²⁹ nano- $\text{SnCl}_4 \cdot \text{SiO}_2$,³⁰ nano- ZnO ,³¹ iodine,³² silica sulfuric acid³³ and $[\text{Et}_3\text{NSO}_3\text{H}]\text{Cl}$ ³⁴ have been used for the preparation of 14-aryl-14*H*-dibenzo[*a,j*]xanthenes. $\text{SbCl}_3/\text{SiO}_2$,³⁵ SiO_2 - RSO_3H ,³⁶ *p*-dodecylbenzenesulfonic acid,³⁷ triethylbenzylammonium chloride³⁸ and diammonium hydrogen phosphate³⁹ have been used for the synthesis of 3,4,5,6,7,9-hexahydro-1*H*-xanthene-1,8(2*H*)-diones octahydroxanthenes. Sulfamic acid,⁴⁰ dodecatungstophosphoric acid,⁴¹ $\text{InCl}_3/\text{P}_2\text{O}_5$ ⁴² and poly(2-acrylamido-2-methylpropane sulphonic acid) crosslinked with *N,N'*-methylenebis(acrylamide) (poly(AMPS-co-AA))⁴³ have been used for the preparation of tetrahydrobenzo[*a*]xanthen-11-ones. However, some of these methods involved long reaction times, harsh reaction conditions, or unsatisfactory yields. Therefore, improvements of these synthesis methods have been continuously sought.

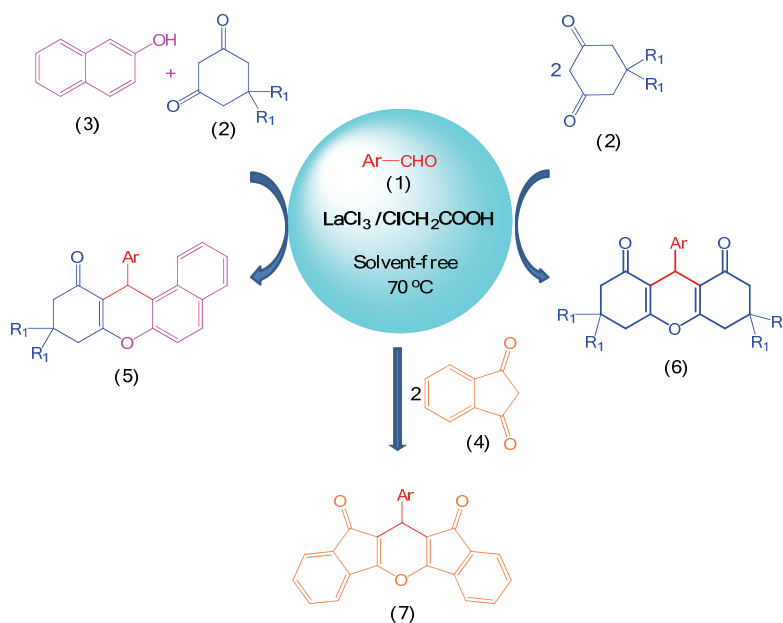
In continuation of ongoing research on green catalytic systems and their applications in organic synthesis and their applications as catalysts in organic synthesis,^{44–46} it was decided to investigate lanthanum(III) chloride/chloroacetic acid ($\text{LaCl}_3/\text{ClCH}_2\text{COOH}$) as a highly efficient, and reusable catalytic system for the practical, one-pot, three-component synthesis of 11*H*-benzo[*a*]xanthen-11-ones, hexahydro-1*H*-xanthene-1,8(2*H*)-diones and 11-aryl-10*H*-diindeno[1,2-*b*:2',1'-*e*]pyran-10,12(11*H*)-diones under solvent-free conditions (Scheme 1).

EXPERIMENTAL

Chemicals and apparatus

Chemicals were either laboratory-prepared or purchased from Merck or Fluka companies, and were used without any further purification.

The IR spectra were recorded in KBr using a Bruker FT-IR spectrophotometer. The ¹H- and ¹³C-NMR were recorded in CDCl_3 and $\text{DMSO-}d_6$ on a Bruker DRX-500 spectrometer using tetramethylsilane as an internal reference. The melting points were determined with a hot-plate microscope apparatus. The purity determination of the substrates and reaction monitoring were accomplished by TLC (petroleum ether (b.p.: 35–60 °C)–ethyl acetate (b.p.: 77.1 °C), 3:1) on silica-gel POLYGRAM SIL G/UV₂₅₄ plates (Merck).



Scheme 1. One-pot synthesis of xanthene derivatives using $\text{LaCl}_3/\text{ClCH}_2\text{COOH}$ as an efficient and reusable catalytic system.

General procedure for the synthesis of 12-aryl-8,9,10,12-tetrahydro-1H-benzo[a]xanthene-11-ones (5)

A mixture of aldehyde (**1**, 1 mmol), β -naphthol (**3**, 1 mmol), dimedone or 1,3-cyclohexanedione (**2**, 1.2 mmol) and $\text{LaCl}_3/\text{ClCH}_2\text{COOH}$ (10 mol %) was stirred in an oil-bath at 70 °C for a reasonable time. After completion of the reaction, as monitored by TLC, the reaction mixture was cooled, diluted with Et_2O (5 mL) and filtered to remove catalyst. The crude product was recrystallized from ethanol to afford the pure product.

General procedure for the synthesis of compounds 6 and 7

To a mixture of aromatic aldehyde (**1**, 1 mmol) and a cyclic 1,3-dicarbonyl compounds (**2** or **4**, 2 mmol), $\text{LaCl}_3/\text{ClCH}_2\text{COOH}$ (10 mol %) was added and the mixture was heated on an oil bath at 70 °C for a reasonable time. After completion of the reaction, hot ethanol was added to the mixture and then filtered. The residue was recrystallized from ethanol.

The spectral data of the synthesized compounds **5** and **6** are presented in the Supplementary material to this paper.

RESULTS AND DISCUSSION

Initially, to achieve suitable reaction conditions, the one-pot, three-component reaction of 2-naphthol, benzaldehyde, dimedone and $\text{LaCl}_3/\text{ClCH}_2\text{COOH}$ as a model reaction was investigated. Subsequently efforts were focused on the optimization of various reaction parameters, such as amount of $\text{LaCl}_3/\text{ClCH}_2\text{COOH}$ and temperature, in terms of yield and time. This observation revealed that the use of 10 mol % $\text{LaCl}_3/\text{ClCH}_2\text{COOH}$ at 70 °C under solvent-free

conditions produced 9,9-dimethyl-12-phenyl-8,9,10,12-tetrahydro-11*H*-benzo[*a*]-xanthen-11-one (**5a**), after 10 min, in 96 % yield. Notably, the desired product could not be obtained under similar reaction conditions, even after a long time (1 h) in the absence of the catalyst (Table I).

TABLE I. Screening of the reaction conditions for the synthesis of 9,9-dimethyl-12-phenyl-8,9,10,12-tetrahydro-11*H*-benzo[*a*]xanthen-11-one (**5a**)

Entry	Amount LaCl ₃ /ClCH ₂ COOH, mol%	Temperature, °C	Time, min	Yield ^a , %
1	No catalyst	70	60	0
2	LaCl ₃ (10 mol %)	50	35	20
3	LaCl ₃ (15 mol %)	60	30	25
4	ClCH ₂ COOH (10 mol %)	70	50	35
5	ClCH ₂ COOH (20 mol %)	80	55	40
6	LaCl ₃ /ClCH ₂ COOH (5 mol %)	70	25	70
7	LaCl ₃ /ClCH ₂ COOH (10 mol %)	60	20	85
8	LaCl ₃ /ClCH ₂ COOH (10 mol %)	70	10	96
9	LaCl ₃ /ClCH ₂ COOH (10 mol %)	80	10	96

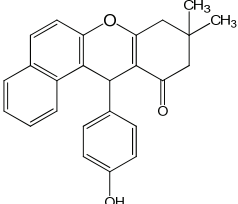
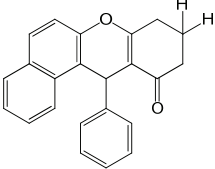
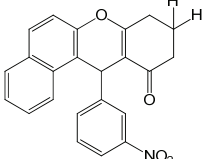
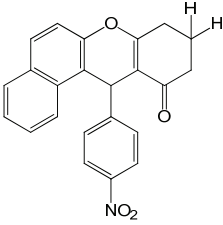
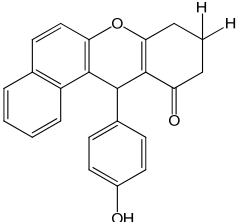
^aIsolated yields

In order to study the generality of the procedure, three series of various xanthen derivatives having different steric and electronic properties were synthesized using the optimized conditions. In all cases, the corresponding products were obtained in good to excellent yields. The results are presented in Tables II and III. The obtained products were characterized by IR, ¹H-NMR and ¹³C-NMR spectroscopy and physical data and the results were compared with the corresponding values for known xanthenes.

TABLE II. One-pot preparation 12-aryl-8,9,10,12-tetrahydrobenzo[*a*]xanthen-11-ones

Entry	X	R	Product	Time, min	Yield, %	M.p. / °C	
						Found	Reported
5a	H	CH ₃		10	96	150–152	151–153 ⁴²

TABLE II. Continued

Entry	X	R	Product	Time, min	Yield, %	M.p. / °C	
						Found	Reported
5b	4-OH	CH ₃		11	90	209–211	209–211 ⁴²
5c	H	H		10	92	189–191	189–190 ⁴⁷
5d	3-NO ₂	H		10	97	233–238	235–236 ⁴⁷
5e	4-NO ₂	H		10	92	234–236	234–235 ⁴⁷
5f	4-OH	H		10	93	267–270	269–270 ⁴⁷

^aIsolated yields

In order to show the merit of the present work, the present results were compared with those reported by other groups in the synthesis of 9,9-dimethyl-12-phenyl-8,9,10,12-tetrahydro-11*H*-benzo[*a*]xanthene-11-one (**5a**). It is important to note that LaCl₃/ClCH₂COOH acts as an effective catalytic system with respect to reaction time and yield (Table IV).

In a plausible mechanism, at first, the carbonyl group of the aromatic aldehyde is activated through coordination with acidic LaCl₃/ClCH₂COOH to give **8**.

Next, the carbonyl carbon is attacked by the nucleophilic 1,3-dione derivative to form Knoevenagel products. The subsequent addition of these fragments to **2–4** gives the acyclic adduct intermediate, which undergoes intramolecular cyclization with participation of two hydroxyl groups to afford the xanthene derivatives (Scheme 2).

TABLE III. One-pot preparation of hexahydro-1*H*-xanthene-1,8(2*H*)-dione and diindeno[1,2-*b*:2',1'-*e*]pyran-diones

Entry	X	R ₁	Product	Time, min	Yield, %	M.p. / °C	
						Found	Reported
6a	H	H		10	86	202–204	201–203 ⁴⁸
6b	4-Cl	H		10	92	232–230	231–233 ⁴⁸
6c	4-OH	H		10	97	243–246	245–247 ⁴⁸

TABLE III. Continued

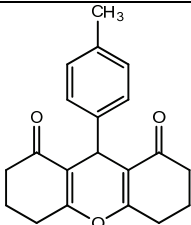
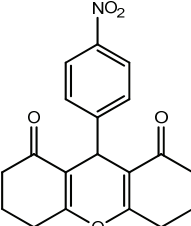
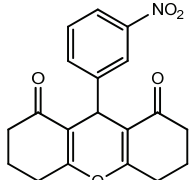
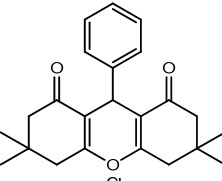
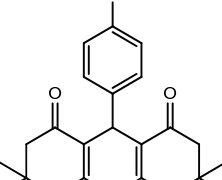
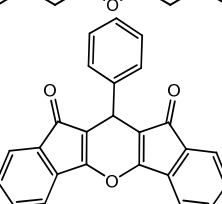
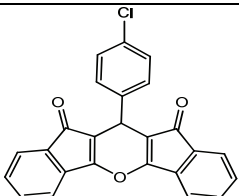
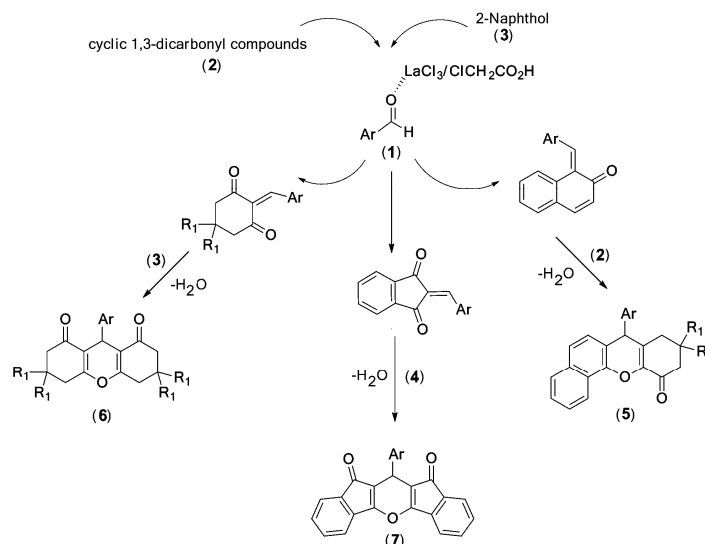
Entry	X	R ₁	Product	Time, min	Yield, %	M.p. / °C	
						Found	Reported
6d	4-CH ₃	H		10	92	217–219	216–218 ⁴⁸
6e	4-NO ₂	H		8	93	245–248	246–248 ⁴³
6f	3-NO ₂	H		7	96	236–240	277–279 ⁴³
6g	H	CH ₃		8	90	202–204	203–204 ⁴⁹
6h	4-Cl	CH ₃		9	93	230–233	230–232 ⁴⁹
7a	H	–		10	80	288–290	290–291 ⁵⁰

TABLE III. Continued

Entry	X	R ₁	Product	Time, min	Yield, %	m.p. / °C	
						Found	Reported
7b	4-Cl	–		9	85	310–312	–

^aIsolated yieldsTABLE IV. Comparison of the results for the synthesis of xanthene **5a** with different catalysts: DBSA, *p*-dodecylbenzenesulfonic acid; TBAHS, tetrabutylammonium hydrogen sulfate; experimental conditions: benzaldehyde (1 mmol), 2-naphthol (1 mmol), dimedone (1 mmol) and LaCl₃/ClCH₂COOH (10 mol %)

Catalyst	Catalyst load mol %	Solvent	Temp., °C	Time, min	Yield ^a %
DBSA	10	H ₂ O, ultrasound	30	60	89 ⁵¹
TMSCl	100	MeCN	reflux	420	95 ⁵²
HClO ₄ -SiO ₂	10	Solvent-free	140	180	32 ⁵³
SbCl ₃ -SiO ₂	10	Solvent-free	120	50	93 ³⁵
PPA-SiO ₂	10	Solvent-free	140	30	93 ⁵³
TBAHS	10	Dioxane, H ₂ O	reflux	210	88 ⁵⁴
LaCl ₃ /ClCH ₂ COOH	10	Solvent-free	70	10	96

^aIsolated yieldsScheme 2. The proposed mechanism for the synthesis of xanthene derivatives using LaCl₃/ClCH₂COOH.

Finally, the possibility of recycling of $\text{LaCl}_3/\text{ClCH}_2\text{COOH}$ was investigated using the model reaction forming **5a** in the presence of $\text{LaCl}_3/\text{ClCH}_2\text{COOH}$. After completion of the reaction, ice-cold water was added to the reaction mixture and the product was filtered. After completely washing the solid product with water, the aqueous layer containing the catalytic system (LaCl_3 and ClCH_2COOH are soluble in water) was evaporated under reduced pressure and catalyst was recovered and reused for subsequent reactions. The recovered catalyst showed the same activity as that of the fresh catalyst without any loss of activity in terms of yield and product purity. The catalyst was recycled and reused in the same reaction at least four times with remarkable retention of its activity (Table V).

TABLE V. Recycling yields (refer to pure isolated yields); reaction conditions: benzaldehyde (1 mmol), 2-naphthol (1 mmol), dimedone (1 mmol) and $\text{LaCl}_3/\text{ClCH}_2\text{COOH}$ (10 mol %)

No. of Cycles	Fresh	Run 1	Run 2	Run 3	Run 4
Yield, %	96	96	96	96	96
Time, min	10	10	10	10	10

CONCLUSIONS

In conclusion, an efficient catalytic system was developed for the synthesis of various xanthene derivatives *via* one-pot three-component reactions of aldehydes, 2-naphthol, and cyclic 1,3-dicarbonyl compounds. The advantages of the presented procedure include simplicity of operation, high yields of products, short reaction time and solvent-free conditions.

SUPPLEMENTARY MATERIAL

Analytical and spectral data of the synthesized compounds are available at the pages of the journal's website: <http://www.shd.org.rs/JSCS/>, or from the corresponding author on request.

Acknowledgement. We gratefully acknowledge the funding support received for this project from the Research Council of Shahid Bahonar University of Kerman.

ИЗВОД

БРЗА СИНТЕЗА КСАНТЕНСКИХ ДЕРИВАТА УПОТРЕБОМ КАТАЛИТИЧКОГ СИСТЕМА ЛАНТАН(III)-ХЛОРИД/ХЛОРСИРЉЕТНА КИСЕЛИНА У ОДСУСТВУ РАСТВОРАЧА

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$\text{LaCl}_3/\text{ClCH}_2\text{COOH}$ је коришћен као ефикасан каталитички систем, који се успешно може поновно користити више пута после изоловања, за синтезу деривата 11H-бензо[a]-ксантен-11-она, хексахидро-1H-ксантен1,8(2H)-диона и 11-арил-10H-дииндено[1,2-b:2',1'-e]пиран-10,12(11H)-диона. Синтеза се врши у једном кораку, у трокомпонентној реакционој смеси алдехида, 2-нафтола, и цикличног 1,3-дикарбонилног једињења.

Реакција се одвија брзо, на 70 °C без присуства растварача а жељени производи се добијају у добром до одличном приносу.

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