Research on Chemical Intermediates
https://doi.org/10.1007/s11164-019-03945-7

Sodium acetate/MWI: a green protocol for the synthesis of tetrahydrobenzo[α]xanthen-11-ones with biological screening

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Received: 9 April 2019 / Accepted: 29 July 2019

Abstract

An efficient, straightforward and rapid method has been developed for the synthesis of tetrahydrobenzo[α] xanthen-11-one using sodium acetate. The reaction was performed via three components: β-naphthol, dimedone with various aromatic aldehydes under solvent-free microwave irradiation and conventional heating condition. We observed an efficient result for microwave synthesis than conventional heating. The synthesized tetrahydrobenzo[α]xanthen-11-one (4a–l) was screened for their antimicrobial activity against two bacteria (*Escherichia coli* and *Bacillus subtilis*) and two fungal strains (*Aspergillus niger* and *Saccharomyces cerevisiae*). The synthesized products: 9, 10-dihydro-9, 9-dimethyl-12-(4-nitrophenyl)-8H-benzo[a] xanthen-11(12H)-one, 9, 10-dihydro-9, 9-dimethyl-12-(2, 4-dichlorophenyl)-8H-benzo[a]xanthen-11(12H)-one, 9, 10-dihydro-9, 9-dimethyl-12-(6-methyl-4-oxo-4H-chromen-3-yl)-8H-benzo[a]xanthen-11(12H)-one exhibit good antimicrobial activity. The developed protocol is graced with appealing advantages such as high purity, simple operation, excellent yields, inexpensive and easy availability of catalyst.

Electronic supplementary material The online version of this article (https://doi.org/10.1007/s11164-019-03945-7) contains supplementary material, which is available to authorized users.

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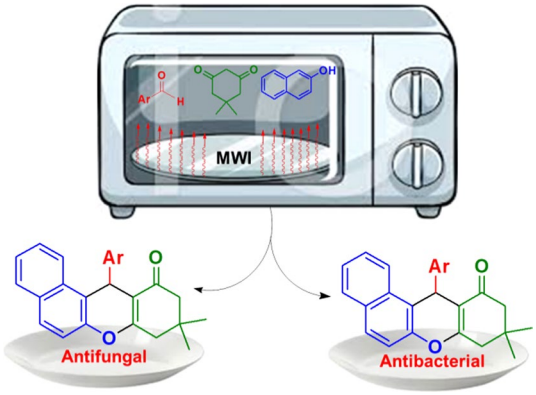
Published online: 05 August 2019

Graphic abstract



Keywords Antimicrobial screening · Microwave irradiation · Solvent-free condition · Tetrahydrobenzo[α] xanthen-11-one

Introduction

Multicomponent reactions (MCRs) grabbed the attention of many researchers due to easy synthesis of biologically active heterocycles [1, 2]. Multicomponent reactions are the important tools in the synthesis of chemical compounds and drugs because they have valuable advantages over conventional synthesis [3]. They show the ideal synthesis of heterocycles with high atom efficiency, simple implementation, environment benign approach and proficient conditions [4, 5]. The development and application of sustainable multicomponent reactions (MCRs) has become an increasingly important topic in chemistry since MCRs allow the sustainable formation of products with high diversity and complexity in a combinatorial one-pot fashion [6, 7].

Recently, the microwave-assisted organic synthesis (MAOS) is one of the successful methodologies for organic transformations. Microwave-assisted organic synthesis shows many advantages including faster, cleaner reactions, atom economy, high product yields and operational simplicity [8–10]. Presently, the green synthesis has become one of the most widely used approaches for the routine synthesis of biologically active heterocyclic derivatives [11, 12]. Thus, the use of MW-assisted synthesis in drug discovery is a large and rapidly growing research field [13, 14].

Xanthenes and benzoxanthenes are privileged group of organic compounds because of their extensive range of biological and pharmaceutical properties [15–17]. Xanthene derivatives could be applied as dyes [18], cytotoxic agents [19], anti-inflammatory [20], antiviral activities [21], bactericidal [22], antagonists to inhibit zoxalamine activity [23], in laser technology [24]. Tetrahydrobenzo[a] xanthen-11-ones specified wide applications in the pharmacological and industrial

sectors. The various methodologies were studied for their synthesis by using three-component condensation of dimedone, aromatic aldehyde and β-naphthol. Some methodologies for the protocol were p-toluene sulfonic acid/ionic liquid, bronsted acidic ionic liquid, proline triflate, ceric ammonium nitrate, iodine and $InCl_3/P_2O_5$ [25–39].

The vital disadvantages of reported methodologies are high-price reagents, long reaction time, tedious work-up, large catalyst loading and hazardous reaction conditions. Hence, we designed a green protocol which diminishes these disadvantages. We developed sodium acetate/MWI as new catalytic system with high catalytic efficiency, short reaction time, high product yield and easy work-up for the synthesis of tetrahydrobenzo[α] xanthen-11-one. This is first time report on tetrahydrobenzo[α] xanthen-11-one green synthesis using sodium acetate/MWI catalytic system.

Experimental

General

Aldehyde, dimedone and β-naphthol were purchased from Sigma-Aldrich and used as received; 3-formyl chromone was prepared by Vilsmeier–Haack reaction of 2-hydroxy acetophenone with DMF and $POCl_3$. Melting points were recorded by open-glass capillary method. The progress of reaction was monitored by thin-layer chromatography (TLC). The IR spectrum (υmax, cm-1) was recorded on lambda FTIR-7600 spectrophotometer using KBr pellets. ^{1}H and ^{13}C spectra of pure products were recorded by using $CDCl_3$ solvent on Brucker 400 MHz and 100 MHz spectrometer, respectively, with TMS as an internal standard. The proposed reaction is conducted in Raga Scientific Microwave.

Typical procedure for the synthesis of tetrahydrobenzo[α]xanthen-11-one derivatives

A mixture of aromatic aldehyde (1 mmol), dimedone (1 mmol) and β-naphthol (1 mmol) was microwave irradiated at 450 W with sodium acetate (15 mol%). The TLC technique (ethyl acetate: hexane—1:9, v/v) was used to monitor the proposed reaction. After the successful completion of reaction, reaction mixture was poured to ice-cold water, resulting into the product. The catalyst sodium acetate becomes soluble in water. The product obtained was recrystallized from ethanol to obtain the pure 9, 10-dihydro-9, 9-dimethyl-12-phenyl-8H-benzo[a]xanthen-11(12H)-one.

Results and discussion

Optimization of reaction conditions

In quest of best reaction condition, we successfully conducted a reaction between an aromatic aldehyde, β-naphthol and dimedone in the presence of sodium acetate under microwave irradiation. The various number of trail reactions between 4-chlorobenzaldehyde (1 mmol), β-naphthol (1 mmol) and dimedone (1 mmol) were carried for optimizing exact reaction condition in the absence or presence of catalyst under microwave irradiation. The said reaction was also performed with conventional heating (Table 1). The efficient results were obtained with 15 mol% sodium acetate under microwave irradiation for tetrahydrobenzo[α]xanthen-11-one synthesis (Table 2).

Initially, we have attempted conventional method by using NaOAc at 80 °C for the synthesis of tetrahydrobenzo[α]xanthen-11-one (Scheme—1, 4a–l). The results are summarized in Table 1. We observed long reaction time for completion of proposed synthesis with low yield.

Under optimized condition, the reaction of 4-chlorobenzaldehyde, β-naphthol and dimedone resulted into the product 9, 10-dihydro-9, 9-dimethyl-12-(4-chlorophenyl)-8H-benzo[a]xanthen-11(12H)-one with 95% yield within 5 min. The various substituted aromatic aldehydes (electron withdrawing, electron donating and 3-formyl chromones) were reacted with β-naphthol and dimedone to check the generality and effectiveness of our developed protocol. These reactions conducted successfully, smoothly and furnished with efficient desired tetrahydrobenzo[α]xanthen-11-one product. The proposed reaction via NaOAc/MWI catalysis is relatively proficient with respect to yield and time of reaction than other reported catalysis (Table 3). Sodium acetate is completely soluble in water; hence, water is used for the complete removal of sodium acetate during work-up. This indicates there is no leaching or degradation behavior during work-up.

Table 1 The optimization of catalyst (sodium acetate) loading for the synthesis of tetrahydrobenzo[α]xanthen-11-one in different reaction mediums

Entry	Catalyst loading (mol%)	Temperature	Time (min)	Yield (%)
1	No catalyst	120	600	Trace
2	5	80	90	20
3	5	100	150	25
4	5	120	480	54
5	10	27	600	35
6	10	120	180	90
7	15	150	360	65
8	20	120	210	90
9	5	MWI	5	65
10	10	MWI	7	72
11	**15**	**MWI**	**5**	**95**

Table 2 Synthesis of various tetrahydrobenzo[α]xanthen-11-ones using NaOAc as a catalyst under microwave irradiation and solvent-free condition

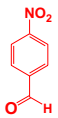
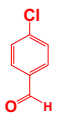
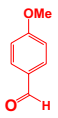
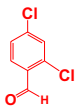
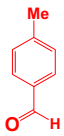
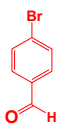
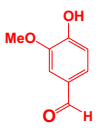
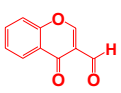
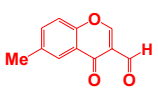
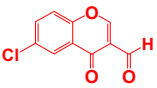
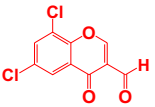
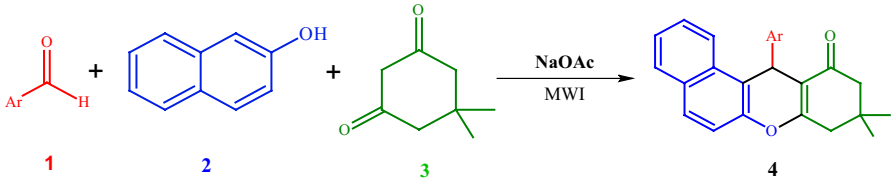
Entry	Aromatic aldehyde	Microwave irradiation		Conventional heating		Melting point (°C)	
		Time (min)	Yield (%)	Time (h)	Yield (%)	Found	Literature
4a		5	92	3.0	90	152–153	151–153 [30]
4b		5	92	5.0	89	177–180	178–180 [30]
4c		5	95	5.5	91	180–182	180–182 [30]
4d		6	88	6.5	85	204–205	204–205 [30]
4e		7	95	5.5	93	176–177	175–177 [31]
4f		5	88	6.5	85	175–177	176–178 [30]
4 g		7	89	6.5	88	187–188	186–188 [30]
4 h		8	88	6.5	86	214–215	213–215 [31]
4i		5	90	7.0	84	215–218	New Report
4j		7	92	7.5	87	220–223	New Report

Table 2 (continued)

Entry	Aromatic aldehyde	Microwave irradiation		Conventional heating		Melting point (°C)	
		Time (min)	Yield (%)	Time (h)	Yield (%)	Found	Literature
4 k		7	85	8.0	82	224–226	New Report
4 l		8	88	8.0	84	239–242	New Report
4 m		6	88	6.0	86	174–176	176–178 [40]
4n		7	89	6.5	85	170–172	170–172 [40]

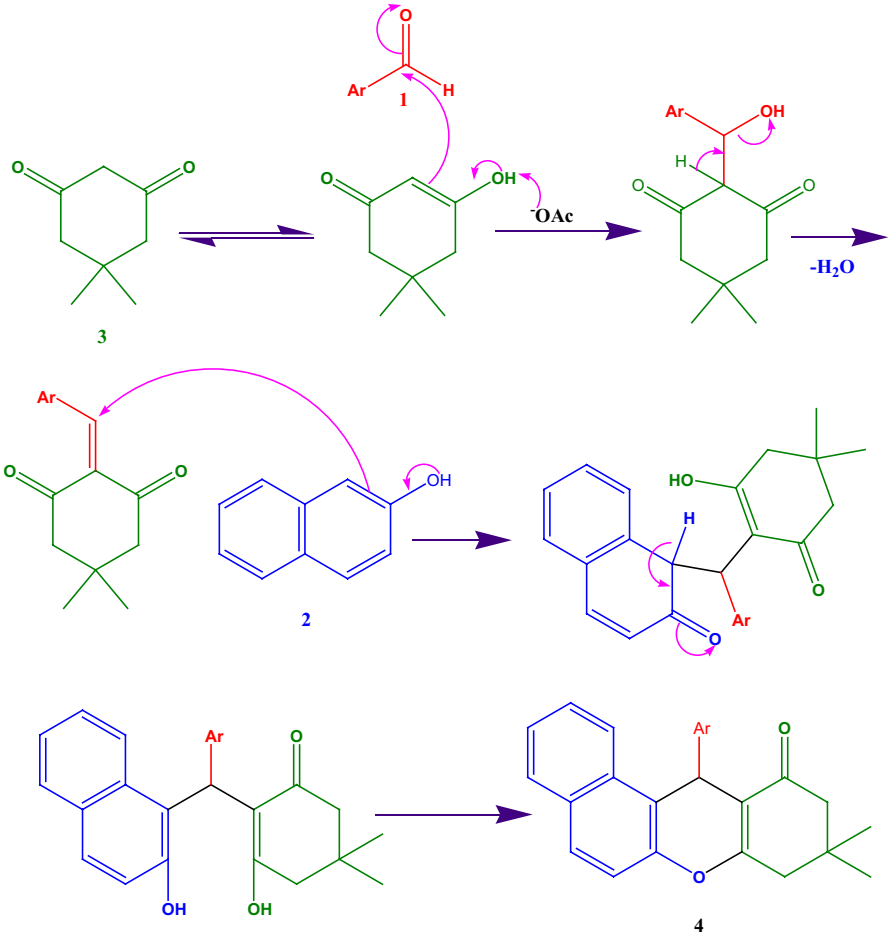


Scheme 1 Multicomponent reaction between aromatic aldehyde (1 mmol), dimedone (1 mmol) and β-naphthol (1 mmol)

Table 3 Synthesis of tetrahydrobenzo[α]xanthen-11-one in the presence of different catalysts

Sr. no.	Catalyst	Conditions	Time (min.)	Yield (%)	Literature
1	$Sr(OTf)_2$	1,2-Dichloroethane, 80 °C	300	85	[35]
2	NH_4Cl	Solvent free, 120 °C	15	86	[36]
3	CAN	Solvent free, 120 °C	30	94	[37]
4	InCl3	Solvent free, 120 °C	30	84	[38]
5	p-TSA	Solvent free, 120 °C	45	88	[39]
6	$H_3PW_{12}O_{40}$	Solvent free, 60 °C	70	86	[41]
7	Graphene oxide	Solvent free, 120 °C	20	85	[42]
8	I_2	Ethanol, reflux	180	84	[43]
9	NaOAc	MWI, 450 W	5	95	Present work

A plausible mechanism of this condensation reaction is revealed in Scheme 2. In the first step, the olefin formation takes place by a Knoevenagel condensation between an aromatic aldehyde and dimedone which is catalyzed by sodium acetate. Further, Michael addition reaction of β-naphthol takes place with olefin intermediate



Scheme 2 A feasible mechanism for the synthesis of tetrahydrobenzo[α]xanthen-11-one

and undergoes dehydration which results into the target molecule tetrahydrobenzo[α] xanthen-11-one.

Minimum inhibitory concentration (MIC) evaluation

The agar cub method [44] imparted the minimum inhibitory concentration (10–100 µg) for antimicrobial testing of various tetrahydrobenzo[α]xanthen-11-one derivatives. The two bacterial strains, *E.coli* (gram-negative) and *B. subtilis* (gram-positive bacteria) were used for the determination of MIC values using ampicillin as positive and 1% DMSO as negative control. Similarly, two fungal stains: *A. niger* and *S. cereviase* using miconazole, as the standard drug was evaluated for the minimum inhibitory concentration. The above data represent the mean ± standard error of three replicates (Table 4). MIC is the lowest concentration of a drug that inhibits the growth of a particular

Table 4 Minimum inhibitory concentration (MIC) evaluation of synthesized various tetrahydrobenzo[α] xanthen-11-one

Entry	Antibacterial activity		Antifungal activity	
	E. coli	*B. subtilis*	*A. niger*	*S. cereviase*
4a	33.00 ± 1.00	12.17 ± 076	19.50 ± 0.50	31.17 ± 0.29
4b	18.17 ± 1.26	20.33 ± 1.53	22.33 ± 0.58	19.33 ± 0.58
4c	30.67 ± 1.53	19.33 ± 1.04	26.17 ± 0.76	14.17 ± 0.29
4d	28.83 ± 1.26	18.00 ± 0.50	16.50 ± 0.87	25.67 ± 0.58
4e	20.67 ± 2.52	16.00 ± 1.00	14.83 ± 0.76	25.67 ± 1.53
4f	44.33 ± 1.15	22.17 ± 0.76	17.17 ± 1.26	24.17 ± 0.29
4g	21.67 ± 1.15	25.71 ± 0.76	48.67 ± 1.15	46.50 ± 0.50
4h	18.67 ± 0.58	25.00 ± 1.00	27.67 ± 0.58	34.33 ± 0.58
4i	32.50 ± 0.50	33.17 ± 0.76	25.83 ± 0.76	25.00 ± 1.00
4j	17.67 ± 1.53	16.50 ± 0.50	35.00 ± 1.00	28.67 ± 0.58
4k	55.33 ± 1.53	61.33 ± 1.53	26.50 ± 0.50	28.67 ± 0.58
4l	23.00 ± 1.00	18.83 ± 0.76	17.67 ± 0.58	19.33 ± 0.58
Ampicillin	19.67 ± 0.29	14.33 ± 0.58	–	–
Miconazole	–	–	16.67 ± 0.58	20.00 ± 1.00

organism under specific condition. Among them, 9, 10-dihydro-9, 9-dimethyl-12-(4-nitrophenyl)-8H-benzo[a]xanthen-11(12H)-one [4b], 9, 10-dihydro-9, 9-dimethyl-12-(2, 4-dichlorophenyl)-8H-benzo[a]xanthen-11(12H)-one [4e], 9, 10-dihydro-9, 9-dimethyl-12-(6-methyl-4-oxo-4H-chromen-3-yl)-8H-benzo[a]xanthen-11(12H)-one [4j] showed best results. Tetrahydrobenzo[α]xanthen-11-ones can be anticipated to gain synergistic effects on biological activities by the presence of nitro and chloro groups (Entries 4b, 4e, 4j).

Spectroscopic data of some synthesized tetrahydrobenzo[α]xanthen-11-one

9, 10-dihydro-9, 9-dimethyl-12-phenyl-8H-benzo[a]xanthen-11(12H)-one [4a]

IR (υ_{max}, cm^{-1}, KBr): 2956 (C-H), 1655 (C=O), 1621(C=C); **^{1}HNMR (400 MHz, $CDCl_3$, δ ppm):** 0.99 (s, 3H), 1.10 (s, 3H), 2.16 (s, 2H), 2.46 (s, 2H), 4.90 (s, 1H), 7.45–7.31 (5H, m), 7.19–7.15 (2H, m), 7.07–7.03 (1H, m), 7.73–7.82 (m, 2H), 8.17 (d, J = 8.1 Hz, 1H); **^{13}CNMR (100 MHz, $CDCl_3$, δ ppm):** 26.72, 28.42, 31.92, 34.78, 41.02, 50.23, 113.56, 116.28, 123.23, 124.17, 125.18, 126.86, 127.63, 128.08, 128.48, 134.09, 144.26, 147.36, 162.48, 196.11. Anal Calcd for $C_{25}H_{22}O_2$: C, 84.72; H, 6.26%. Found: C, 84.68; H, 6.31%. These spectroscopic values are shown in electronic supplementary material (ESM 1).

9, 10-dihydro-9, 9-dimethyl-12-(4-chlorophenyl)-8H-benzo[a]xanthen-11(12H)-one [4c]

IR (υ_{max}, cm^{-1}): 2952 (C-H), 1652 (C=O), 1621(C=C); **^{1}HNMR (400 MHz, $CDCl_3$, δ ppm):** 1.00 (s, 3H), 1.19 (s, 3H), 2.13 (s, 2H), 2.59 (s, 2H), 5.58 (s, 1H), 6.98 (d, J = 8.2 Hz, 2H), 7.06 (s, 2H), 7.20 (d, J = 8.1 Hz, 1H), 7.31-7.36 (m, 1H), 7.66 (d, J = 8.1 Hz, 1H), 7.78 (d, J = 6.3 Hz, 1H), 7.89 (d, J = 8.1 Hz, 1H); **^{13}CNMR (100 MHz, $CDCl_3$, δ ppm)**: 26.43, 27.72, 30.37, 39.08, 43.80, 51.40, 109.08, 118.98, 122.11, 122.58, 123.12, 123.38, 125.28, 126.08, 127.61, 129.42, 129.71, 131.19, 133.19, 139.61, 151.21, 162.20, 196.98. Anal Calcd for $C_{25}H_{21}ClO_2$: C, 77.21; H, 5.44%. Found: C, 77.23; H, 5.49%. These spectroscopic values are shown in electronic supplementary material (ESM 2).

9, 10-dihydro-9, 9-dimethyl-12-(4-nitrophenyl)-8H-benzo[a]xanthen-11(12H)-one [4b]

IR (υ_{max}, cm^{-1}, KBr): 2953 (C-H), 1648 (C=O), 1622 (C=C), 1532, 1341 (NO_2); **^{1}HNMR (400 MHz, $CDCl_3$, δ ppm):** 1.10 (s, 3H), 1.21 (s, 3H), 2.03 (s, 2H), 2.51 (s, 2H), 5.43 (s, 1H), 7.10–7.32 (m, 4H), 7.58–8.08 (m, 4H), 8.25–8.30 (d, 3H); **^{13}CNMR (100 MHz, $CDCl_3$, δ ppm):** 26.72, 27.43, 33.37, 39.78, 43.29, 51.43, 109.39, 118.88, 121.61, 122.28, 122.12, 123.38, 125.28, 126.08, 127.61, 128.32, 129.21, 135.11, 149.69, 153.21, 162.22, 196.68. Anal Calcd for $C_{25}H_{21}NO_4$: C, 75.17; H, 5.30; N, 3.51%. Found: C, 75.22; H, 5.27; N, 3.55%. These spectroscopic values are shown in electronic supplementary material (ESM 3).

9, 10-dihydro-9, 9-dimethyl-12-(6-methyl-4-oxo-4H-chromen-3-yl)-8H-benzo[a] xanthen-11(12H)-one [4j]

IR (υ_{max}, cm^{-1}, KBr): 2953 (C-H), 1679 (C=O), 1655 (C=O), 1621 (C=C); **^{1}HNMR (400 MHz, $CDCl_3$, δ ppm):** 0.98 (s, 3H), 1.11 (s, 3H), 2.20 (s, 2H), 2.53 (s, 2H), 5.60 (s, 1H), 6.81–6.93 (m, J = 6.4 Hz, 4H), 6.93 (d, J = 8.1 Hz, 1H), 7.13–7.31 (m, 3H), 7.43–7.63 (m, 3H); **^{13}CNMR (100 MHz, $CDCl_3$, δ ppm):** 24.74, 26.92, 27.47, 31.97, 35.78, 42.22, 51.13, 115.36, 116.18, 117.36, 123.08, 123.11, 124.28, 125.28, 126.48, 127.08, 128.68, 128.86, 134.12, 149.39, 154.26, 164.28, 191.18, 197.48. Anal Calcd for $C_{29}H_{24}O_4$: C, 79.80; H, 5.54%. Found: C, 79.78; H, 5.51%. These spectroscopic values are shown in electronic supplementary material (ESM 4).

Conclusion

We explored sodium acetate/MWI as an efficient, rapid green protocol for the synthesis of a wide range of pharmaceutically interesting functionalized tetrahydrobenzo[α] xanthen-11-ones via three-component condensation of dimedone, β-naphthol and various aromatic aldehydes. Tetrahydrobenzo[α]xanthen-11-ones were evaluated for their potent antimicrobial activity. The synthesized products 9, 10-dihydro-9,

9-dimethyl-12-(4-nitrophenyl)-8H-benzo[a]xanthen-11(12H)-one [4b], 9, 10-dihydro-9, 9-dimethyl-12-(2, 4-dichlorophenyl)-8H-benzo[a]xanthen-11(12H)-one [4e], 9, 10-dihydro-9, 9-dimethyl-12-(6-methyl-4-oxo-4H-chromen-3-yl)-8H-benzo[a]xanthen-11(12H)-one [4j] exhibited excellent antimicrobial activity. The noteworthy advantages of present protocol are high yield, shorter reaction time, greener conditions and easy work-up.

Acknowledgements Authors are thankful to Department of Chemistry, Yashavantrao Chavan Institute of Science, Satara, for providing laboratory facilities and also thankful to DBT-STAR Scheme, Research Advisory Committee for giving seed money.

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