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Synthesis of 1, 8-dioxodecahydroacridines via Hantzsch condensation using theophylline in an aqueous medium: an eco-friendly and bio-based approach

Nilam S. Dhane[1] · Aboli C. Sapkal[2] · Suraj R. Attar[2] · Sanchita M. Dhumal[1] · Ganesh K. Chougule[1] · Samadhan P. Pawar[1] · Santosh B. Kamble[2] · Kishor V. Gaikwad[1]

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Abstract
In this work, we explored the convenient and sustainable method for the synthesis of 1, 8 dioxodecahydroacridine derivatives was achieved via a one-pot condensation reaction of dimedone, para-nitrobenzaldehyde, and ammonium acetate by using theophylline as a catalyst at room temperature in aqueous medium. This environmentally friendly method boasts several notable features, including high product yields, rapid completion of reactions, the use of eco-friendly and bio-based catalysts, straightforward work-up procedures without the need for column chromatography, cost-effectiveness, clean synthesis practices that avoid the use of harmful organic solvents, and exceptional atom efficiency.

Keywords Heterocycles · Eco-friendly and bio-based catalyst · 1, 8 Dioxodecahydroacridine · Aqueous medium

Introduction

The growing volumes of waste and the harmful residues that inevitably lead to chemical pollution need to be prevented from entering our environment. Consequently, synthetic chemists are driven to develop safer technologies that are

✉ Santosh B. Kamble
santosh.san143@gmail.com

✉ Kishor V. Gaikwad
kishuvgaikwad@rediffmail.com

Nilam S. Dhane
nilamdhane1996@gmail.com

[1] Rajarshi Chhatrapati Shahu College, Kolhapur, India

[2] Yashavantrao Chavan Institute of Science, Satara, Lead College of Karmaveer Bhaurao Patil University, Satara, India

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crucial for the principles of green chemistry. The development of inventive chemical processes should align with green principles, which encompass practices such as efficient separation and catalyst reutilization, non-toxicity, non-flammability, and the use of environmentally friendly solvents. Water is a solvent that is more readily available, sustainable, and environmentally advantageous [1]. The integration of Multicomponent reactions (MCRs) with the class of heterocyclic chemistry stands as a highly significant notion in organic synthesis [2]. The efficacy of MCRs becomes apparent when applied to generate a range of heterocyclic frameworks [3]. As a result, the advancement of MCRs has emerged as a dynamic and intricate subject in contemporary organic chemistry. This evolution offers a more atom-efficient pathway to access a diverse array of compounds [4]. Heterocyclic compounds hold a noteworthy and distinctive position within the field of medicinal chemistry [5].

A nitrogen-based heterocyclic compound plays a pivotal role, found in a diverse array of biologically active substances. These cores are present either through synthetic means or naturally occurring processes, with a multitude of biological applications [6]. Acridine analogs have a wide range of medicinal applications, as depicted in (Fig. 1) [7] including anti-bacterial (S-303) [8], anti-HIV (CGP40336A) [9], antimalarial (Quinacrine) [10], anti-cancer chemotherapeutics (*N*-(2-(dimethylamino) ethyl)acridine-4-carboxamide (DACA), nitracrine, amsacrine [7], anti-tumor [11], anti-glaucoma [12], and laser dyes due to their high fluorescence efficiency [11].

Hence, the synthesis of derivatives of Acridine diones stands as a crucial and fundamental objective within the field of organic chemistry. Acridine molecule was synthesized by many different catalysts such as, $[MIMPS]_3PW_{12}O_{40}$ (a) and

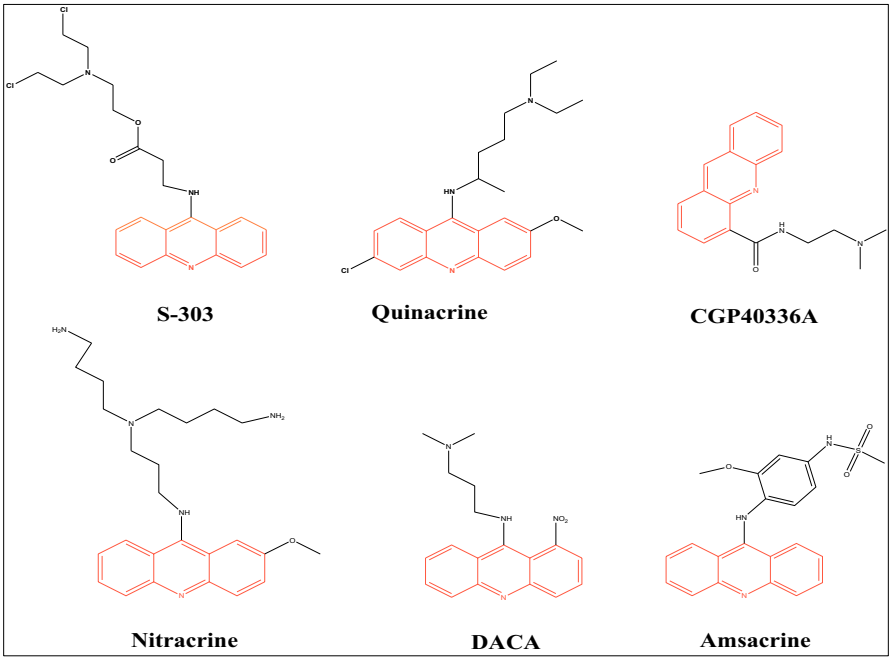


Fig. 1 Biological scaffolds of Acridine derivatives

$[TEAPS]_3PW_{12}O_{40}$ (b) [13], DBH or DCH [14], β-CD-mono SO_3H [15], Nano-FGT [16], Cobalt–alanine metal complex [17], Mo_{132} [18], $[TBA]_2[W_6O_{19}]$ [19], Melamine—formaldehyde resin supported H^+ (MFRH) [20], Green graphene oxide incorporated strontium magnetic nanocatalyst (MSrGO NCs) [21], Tungstophosphoric acid nanoparticles supported on polyamic acid (TPA NPs/PAA) [22], Na^+-MMT-[bip]-NH_2^+ HSO_4^- [23], Molybdic acid-functionalized silica-coated nano-Fe_3O_4 particles (Fe_3O_4@SiO_2–MoO_3H) [24], Ferric hydrogen sulfate supported on silica-coated nickel ferrite.

Nanoparticles [$NiFe_2O_4$@SiO_2-FHS] [25], Cerium-doped ZSM-5 [26], Carbon-doped MoO_3 [27], Dicationic ionic liquid [28], KCC-1-*nPr*-NH-Arg [29]. Frequently, they suffer from the disadvantages of extended reaction duration, harsh reaction conditions, toxicity, and challenges in isolating the final products. These factors restrict their applicability in the construction of intricate molecules. Drug molecules have significant attention in research because of their eco-friendly and bio-based properties [30]. Numerous benefits come with utilizing theophylline (Fig. 2) as an environmentally friendly and bio-based catalyst [31].

The benefits of employing theophylline as an eco-friendly and bio-based catalyst in organic compounds synthesis include its easy handling, eco-friendliness, affordability, gentle nature, widespread availability, non-harmful properties, and biodegradability. In this work we introduce the greener synthetic method by reporting a greener, simple, environmental friendly, and mild reaction conditions for the synthesis of 1, 8 dioxodecahydroacridine using theophylline as a green, efficient and bio-based catalyst via Hantzsch condensation. Theophylline is a medication belonging to the methylxanthine class, prescribed for the treatment of respiratory conditions like chronic obstructive pulmonary disease (COPD), wheezing, shortness of breath Emphysema, and other lung diseases, and it is marketed under various brand names [32]. In this work, we would like to report a new greener methodology for the synthesis of 1, 8 dioxodecahydroacridine by using para-nitrobenzaldehyde, dimedone, and ammonium acetate catalyzed by theophylline as an efficient and solid base catalyst in aqueous medium at room temperature.

Result and discussion

We began by assessing the catalytic performance of theophylline in a model system, a three-component reaction involving a mixture of para-nitrobenzaldehyde (1 mmol), dimedone (2 mmol), and ammonium acetate (1 mmol). We fine-tuned the reaction conditions by experimenting with different solvents, and amounts of catalyst. When the reaction was performed without a catalyst at room temperature by using a mixture of

Fig. 2 Structure of theophylline

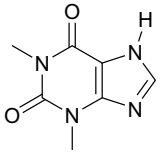
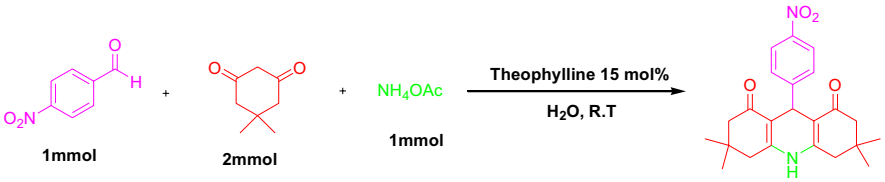


Table 1 Screening of conditions for the synthesis of 1, 8 dioxodecahydroacridine

Entry	Theophylline (mol%)	Solvent/conditions	Time (min)	Isolated yields (%)
1	Catalyst free	H_2O:EtOH, rt	80	Trace
2	5	EtOH, rt	40	60
3	5	H_2O, rt	45	57
4	10	H_2O, rt	25	75
5	10	EtOH, rt	34	82
6	10	H_2O, rt	45	90
7	15	EtOH, rt	17	95
8	15	H_2O, rt	10	98

Reaction conditions: Para-nitrobenzaldehyde (1 mmol), Dimedone(2 mmol), Ammonium acetate (1 mmol)

The yields are related to the isolated products



Scheme 1. Synthesis of 1, 8 dioxodecahydroacridine

solvents H_2O: EtOH for approximately 80 min, only a trace amount of product was obtained (Table 1, entry 1). The most favorable outcome was achieved when we conducted the reaction using theophylline (15 mol%) in a mixture of para-nitrobenzaldehyde (1 mmol), dimedone (2 mmol), and ammonium acetate (1 mmol) in an aqueous medium at room temperature (Table 1, entry 8).

Subsequently, we applied the optimized conditions to the model scheme for the synthesis of 1, 8 dioxodecahydroacridine (Scheme 1), using 15 mol% of theophylline in aqueous medium at room temperature, for the condensation reaction of aromatic aldehydes containing electron-withdrawing and electron-donating groups to synthesize the 1, 8 dioxodecahydroacridine. In addition, turn over number (TON) and turn over frequency (TOF) were calculated for evaluate the efficiency and activity of the catalyst. (Table 2).

To assess how theophylline functions as a catalyst, we have depicted the general schematic representation in Fig. 3, according to the literature [32]. Based on the proposed mechanism theophylline abstracts the proton of active methylene from dimedone then Knoevenagel condensation between dimedone and para-nitrobenzaldehyde to form an intermediate (I). Which further shows Michael addition with another molecule of dimedone to form an intermediate (II). That intermediate (II) again reacts with ammonium acetate to form an intermediate (III) which undergoes intramolecular cyclisation to get the final product (IV) (Fig. 4).

Table 2 Synthesis of 1, 8 dioxodecahydroacridine derivatives catalyzed by theophylline

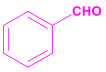
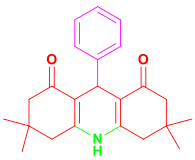
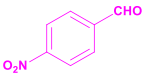
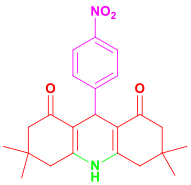
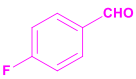
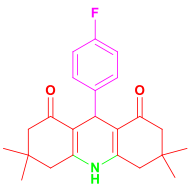
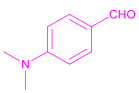
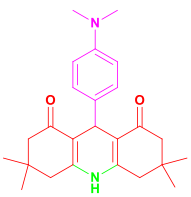
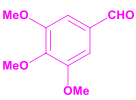
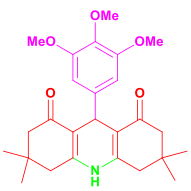
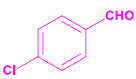
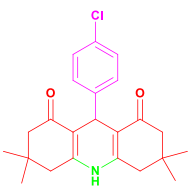
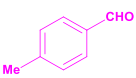
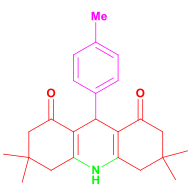
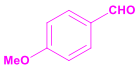
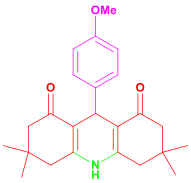
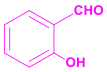
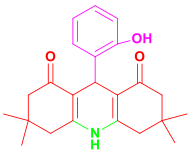
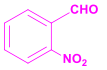
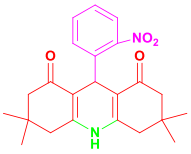
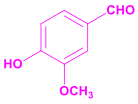
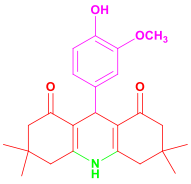
Entry	Aldehyde (1 mmol)	Product	M.P (Obs.)°C	M.P (Theo.)°C	Yield %	TON[$]	TOF*
1			194–196	192–194 [33]	94	6.31	25.24
2			284–286	286–289 [34]	98	6.55	40.93
3			288–290	291–294 [35]	95	6.38	22.78
4			294–296	297–299 [36]	96	6.45	24.80
5			248–250	250–252 [37]	90	6.005	20.01
6			288–289	300–302 [38]	93	6.29	31.45
7			276	279 [39]	89	6.06	28.85

Table 2 (continued)

Entry	Aldehyde (1 mmol)	Product	M.P (Obs.)°C	M.P (Theo.)°C	Yield %	TON[$]	TOF*
8			265	267 [40]	86	5.82	32.38
9			286–288	300 [23]	89	6.05	24.23
10			295–296	297–299 [41]	92	6.20	22.15
11			292–294	294–295 [42]	94	6.38	24.54

Reaction conditions: Para-nitrobenzaldehyde (1 mmol), Dimedone(2 mmol), Ammonium acetate (1 mmol), Theophylline (15 mol%), Distilled water (5 ml)

[$]TON is the moles of reactant converted per mole of catalyst

*TOF was defined as $mol_{product}\ mol^{-1}{}_{catalyst}\ h^{-1}$

Recyclability of catalyst

The primary factor of greatest significance in organic transformations is the ability of a catalyst to be reused efficiently. After completion of the reaction, filter the product which is in crude solid form, and give washings by using distilled water. Then collect the filtrate to recover the catalyst, which is soluble in water, the filtrate was subjected to extraction with diethyl ether. The aqueous layer containing theophylline was separated, and its solvent was removed under reduced pressure to recover and reuse the theophylline. As depicted in Fig. 5, the catalyst recovered from this process exhibited consistent performance up to the second run. However, in the third, fourth, and fifth runs, there was a slight decrease in product yield, possibly attributable to a minor loss of catalyst weight during each recovery cycle. Initially, our focus was on synthesizing 1, 8 dioxodecahydroacridine derivatives, employing theophylline as a highly effective, environmentally friendly, and bio-based catalyst. Here, we present a comparative table that illustrates the differences between the previous study and

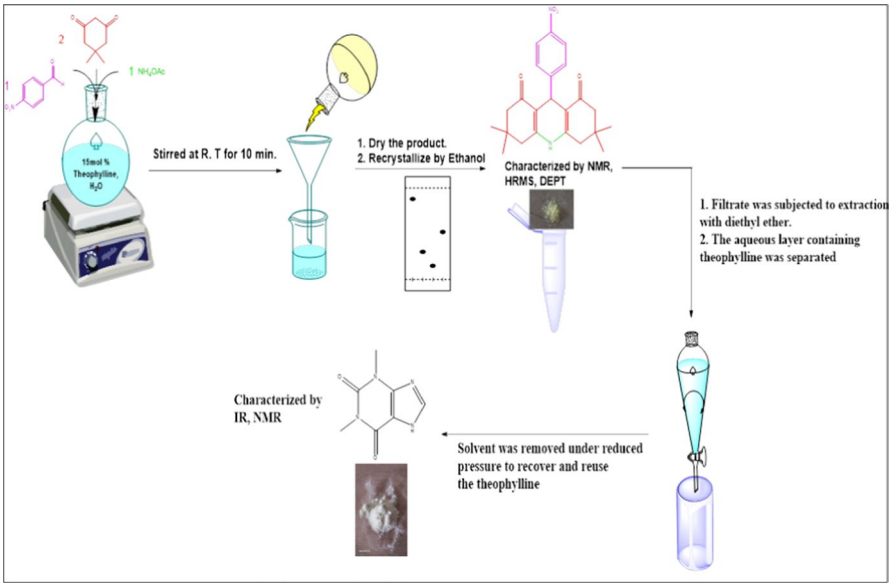


Fig. 3 General schematic representation for the synthesis of 1, 8 dioxodecahydroacridine catalyzed by theophylline in an aqueous medium

the current one (Table 3). In addition after the fifth run both IR and NMR data were recorded. IR data shows that small change in IR (Fig. 6) but NMR data does not show any change (Fig. 7). These findings indicate that initial characteristics of theophylline remained as it is even after fifth run without any change.

Conclusion

In conclusion, using theophylline as a catalyst in the three-component Hantzsch condensation reaction to produce 1, 8 dioxodecahydroacridine presents a compelling eco-friendly and bio-based approach in organic synthesis. The highlighted advantages of this method, such as its environmentally benign reaction conditions, facile product isolation, impressive yields, reduced reaction times, and catalyst recyclability, underscore its alignment with the core tenets of green chemistry. However, as with any novel synthetic approach, several future challenges and opportunities for further improvement can be anticipated. We are exploring a wider range of substrates and functional groups to enhance the versatility and applicability of this method. Investigating methods to improve the catalytic efficiency of theophylline and optimizing reaction conditions for even higher yields, selectivity, and transitioning from laboratory-scale synthesis to larger-scale production while maintaining the efficiency and sustainability of the process for industrial implementation. Delving deeper into the mechanistic aspects of the reaction to gain a comprehensive understanding and potentially refine the reaction pathway. Evaluating the cost-effectiveness of this approach concerning large-scale production and its economic feasibility

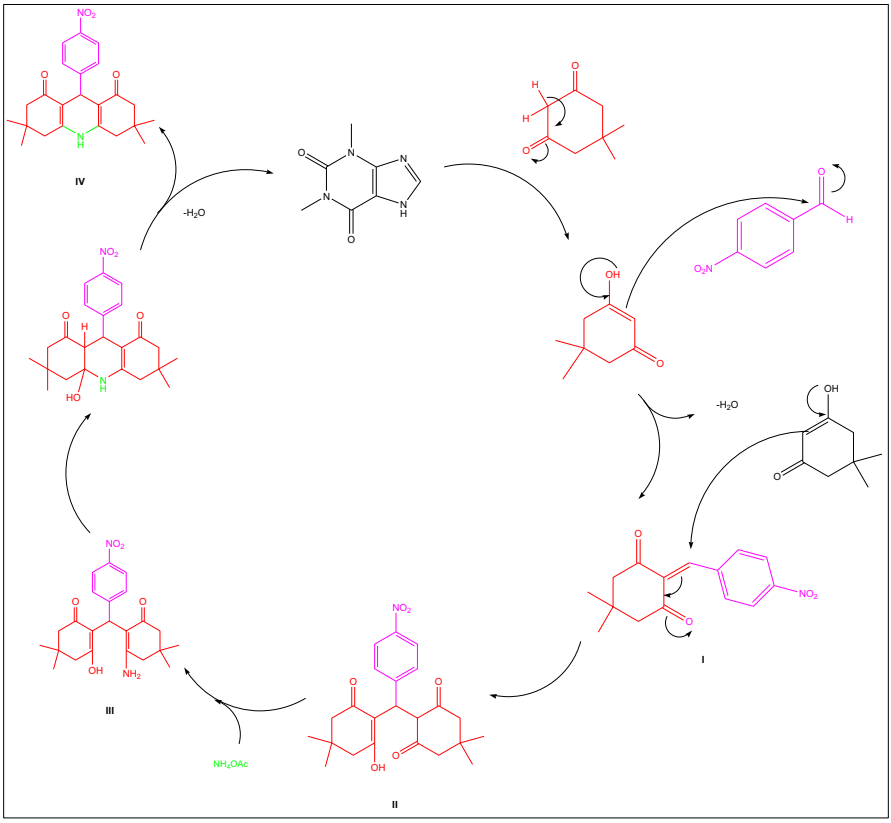


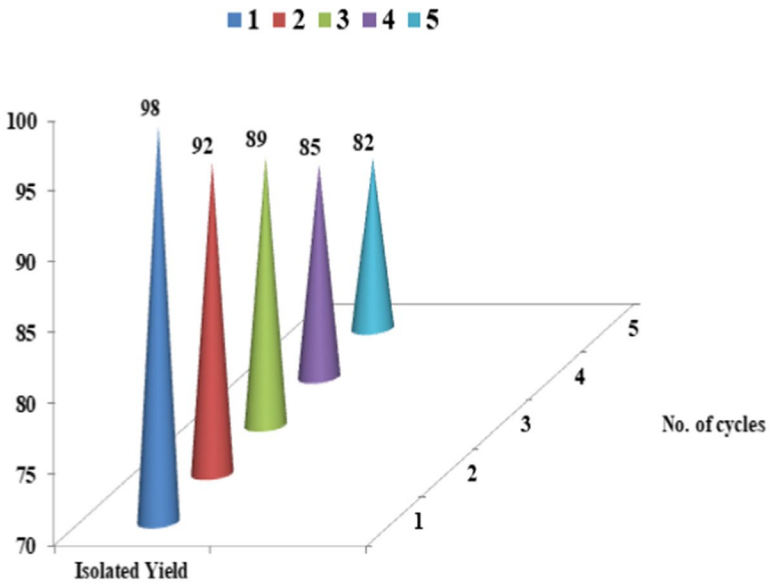
Fig. 4 A plausible reaction mechanism for the synthesis of 1, 8 dioxodecahydroacridine catalyzed by theophylline in an aqueous medium

compared to traditional methods. Addressing these challenges will not only refine this eco-friendly synthetic route but also contribute to its broader adoption in practical applications. By overcoming these hurdles, the synthesis of 1, 8 dioxodecahydroacridine via Hantzsch condensation using theophylline can further solidify its place as a sustainable, efficient, and environmentally benign protocol in the realm of organic synthesis.

Experimental

General

All chemicals were garnered from Loba and Sigma-Aldrich chemical companies and used without any additional purification. Double distilled water was employed as an aqueous medium. The rate of reaction was enhanced by using



No. of Cycles	1	2	3	4	5
Isolated Yield	98	92	89	85	82

Fig. 5 Recyclability of Theophylline

theophylline. Melting points were ruled uncorrected using a melting/boiling point apparatus (EQ730A-EQUIPTRONICS). By using a Bruker or JEOL spectrometer, the ^{1}H and ^{13}C NMR spectra were recorded at 400 and 100 MHz, respectively. High-resolution mass spectra were recorded via a Waters Synapt G2 applying electrospray ionization (ESI).

General procedure for the synthesis of 1, 8 dioxodecahydroacridine

An equimolar mixture of para-nitrobenzaldehyde (1 mmol–0.1511 gm), dimedone (2 mmol–0.5606 gm), ammonium acetate (1 mmol–0.07708 gm), and 15 mol% theophylline (15 mol%–0.027 gm)and double distilled water was stirred under a magnetic stirrer at room temperature and within 10 min we got the crude product. Completion of the reaction was supervised by TLC in n-hexane: ethyl acetate (7:3). The solid product was separated by simple filtration by giving washings of water. The separated solid product was recrystallized by ethanol and characterized by IR, NMR, and HRMS.

1. 9-(4-Nitrophenyl)-3,3,6,6-tetramethyl-1,8-dioxodecahydroacridine (Table 2, Entry-2)

Table 3 A comparative study with Reported reaction conditions

Sr. No	Catalyst	Conditions	Time	Yield (%)	References
1	Chitosan-SO_3H	R. T. H_2O:EtOH	3–12 min	70–95	[43]
2	Ag@n-HA@SiO_2-TAA	Reflux, H_2O	1 h	97	[44]
3	$Ni_{0.5}Co_xZn_{0.5-x}Fe_{2-y}Cr_yO_4$ nanoparticles	Reflux, EtOH	80	84	[45]
4	TiO_2-[bip]-NH_2^+ $C(NO_2)_3^-$	120 °C, Solvent free	12	95	[46]
5	Ru-catalyst	Reflux, tBuOk (25 mol %)	36 h	45–75	[47]
6	Nanostructured molten salt {[HMIM]$C(CN)_3$}	R. T., Solvent free	3 min	98	[48]
7	Amine-functionalized graphene oxide nanosheets (AFGONs)	R. T., EtOH	3 h	86–92	[49]
8	KH_2PO_4	120 °C, EtOH:H_2O	5 h	83	[41]
9	Cellulose[2-(sulfooxy)ethyl]mercaptosulfonic acid[SEMSA]	80 °C, EtOH	10 min	84	[50]
10	Magnetic chitosan-terephthaloyl-creatine bionanocomposite	80 °C, EtOH	1 h 55 min–2 h 20 min	90–95	[51]
11	Fe_3O_4@MgO MNP's	70 °C, Solvent free	5–18 min	89–98	[52]
12	Theophylline	R. T., H_2O	10 min	96–98	Present Work

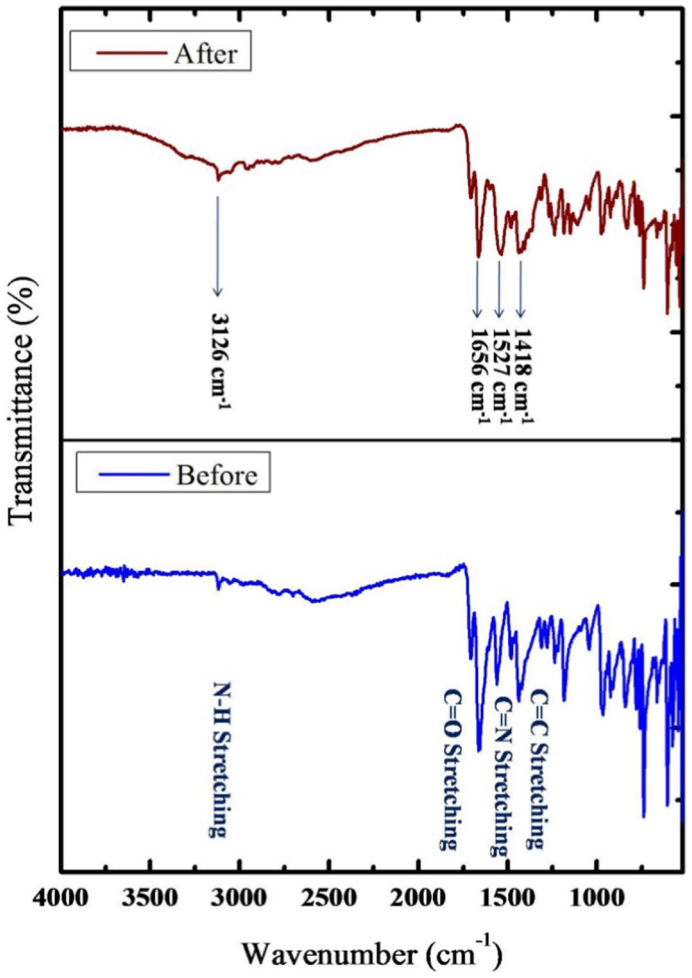


Fig. 6 FTIR of theophylline catalyst before (blue) and after (red) the 5th run of the reaction. (Color figure online)

^{1}H NMR(400 MHz, $CDCl_3$, δ ppm): 1.11 (*s*, 6H), 1.24 (*s*, 6H), 2.36–2.47 (*m*, 8H), 5.54 (*s*, 1H), 7.24–7.26 (*d*, 2H), 8.12–8.14 (*d*, 2H), 11.80 (*s*, 1H); ^{13}C NMR(100 MHz, $CDCl_3$, δ ppm):27.47, 29.53, 31.49, 33.27, 46.41, 46.99, 114.91, 123.32, 127.67, 146.58, 190.97; HR-MS: 395.1971 (M + 1). DEPT of two $-CH_2$ carbon appeared at 46.41 and 47.00 resp.

2. 9-(4-Chlorophenyl)-3,3,6,6-tetramethyl-1,8-dioxodecahydroacridine (Table 2, Entry-6)

^{1}H NMR (400 MHz, $CDCl_3$, δ ppm): 1.09 (*s*, 6H), 1.21 (*s*, 6H), 2.32–2.43 (*m*, 8H), 5.46 (*s*, 1H), 7.00–7.02 (*d*, 2H), 7.21–7.23 (*d*, 2H), 11.87 (*s*, 1H); ^{13}C NMR

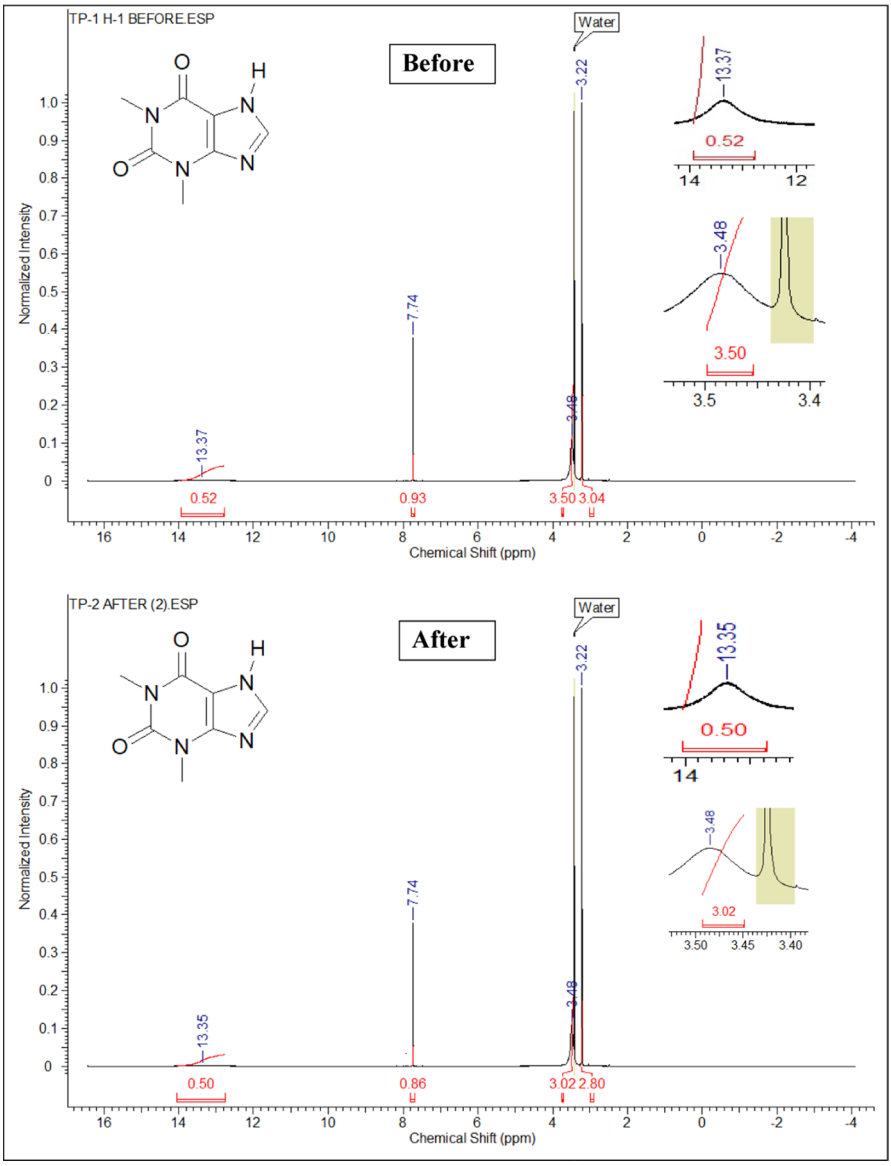


Fig. 7 ^{1}H NMR spectrum of Theophylline (before the use of catalyst) and (after the 5th run of catalyst)

(100 MHz, $CDCl_3$, δ ppm): 27.44, 29.60, 31.44, 32.42, 46.44, 47.06, 115.33, 128.24, 128.35, 131.55, 136.77, 190.65; DEPT of two $-CH_2$ carbon appeared at 46.44 and 47.06 resp.

3. 9-(4-Methoxyphenyl)-3,3,6,6-tetramethyl-1,8-dioxodecahydroacridine (Table 2, Entry-8)

^{1}H NMR(400 MHz, $CDCl_3$, δ ppm): 1.09 (*s*, 6H), 1.22 (*s*, 6H), 2.34–2.42 (*m*, 8H), 3.76 (*s*, 3H), 5.48 (*s*, 1H), 6.79–6.81 (*d*, 2H), 6.99–7.01 (*d*, 2H), 11.92 (*s*, 1H); ^{13}C NMR(100 MHz, $CDCl_3$, δ ppm):27.42, 29.67, 31.42, 32.06, 46.46, 47.10, 55.20, 113.67, 115.80, 127.82, 129.87, 157.62, 190.41.

4. 9-(4-Methylphenyl)-3,3,6,6-tetramethyl-1,8-dioxodecahydroacridine (Table 2, Entry-7).

^{1}H NMR(400 MHz, $CDCl_3$, δ ppm): 0.86 (*s*, 6H), 1.00 (*s*, 6H), 1.97 (*d*, 2H), 2.16 (*d*, 5H), 2.31 (*d*, 2H), 2.44 (*d*, 2H), 4.77 (*s*, 1H), 6.95 (*d*, 2H), 7.04 (*d*, 2H), 9.21 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$, δ ppm): 20.5, 26.4, 29.1, 32.1, 32.3, 50.2, 111.6, 127.5, 128.0, 134.2, 144.3, 149.0, 194.2.

5. 9-(2-Nitrophenyl)-3,3,6,6-tetramethyl-1,8-dioxodecahydroacridine (Table 2, Entry-10).

^{1}H NMR(400 MHz, $CDCl_3$, δ ppm): 0.85 (*s*, 6H), 1.00 (*s*, 6H), 1.93 (*d*, 2H), 2.14 (*d*, 2H), 2.32 (*d*, 2H), 2.45 (*d*, 2H), 5.62 (*s*, 1H), 7.26 (*t*, 1H), 7.34 (*d*, 1H), 7.52 (*t*, 1H), 7.71 (*d*, 1H,), 9.34 (*s*, 1H); ^{13}C NMR(100 MHz, $CDCl_3$, δ ppm): 27.1, 29.3, 29.9, 32.5, 50.7, 111.6, 124.1, 127.0, 130.8, 133.0, 142.2, 148.6, 150.3, 194.6.

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Data availability Data will be made available on request.

Declarations

Conflict of interest The author' declared that they have no conflict of interest.

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