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Cu(II) Schiff Base as Catalyst in the Synthesis of 1,8-Dioxodecahydroacridine

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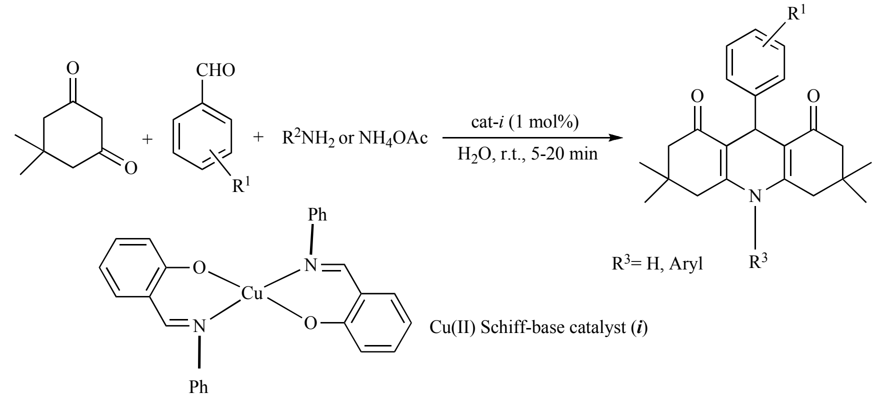
Abstract: The catalytic property of Cu(II) Schiff-base complex in an efficient synthesis of 1,8-dioxodecahydroacridines was investigated. The one-pot three component reaction of dimedone, aromatic aldehydes and aromatic amines or ammonium acetate in water afforded the corresponding 1,8-dioxodecahydroacridines with excellent yields. This reaction was carried out in the presence of 1 mol% of catalysts at room temperature. The reusability of the catalysts was demonstrated by a five-run test without loss of its activity. Also, this catalyst possesses several advantages including mild reaction conditions, lower catalytic loading, shorter reaction times, high yield of the products, inexpensive and cleaner (Green chemistry) reactions.



Keywords: 1,8-Dioxodecahydroacridine, Cu(II) Schiff-base catalyst, multi component reaction, one-pot synthesis, water solvent.

INTRODUCTION

Acridine and its derivatives are interesting compounds, because of their well-known properties in fields of medicinal [1, 62] and biological activities (such as antibacterial [3], antimalarial [4], anticancer [5], antitumor [6], analgesic [7], anticonvulsant [8], hypertensive and anti-inflammatory [9]), applications in material science (semiconductors) [10] and spectroscopy (luminescent agent) [11]. Therefore, their syntheses have been attracted by a large number of organic chemists [12, 13]. The preparation of acridine and its derivatives is a three component reaction that is an important class in organic reaction. The dimedone, aldehyde and primary amine or ammonium acetate are the components of this reaction (Scheme **1**).

Extensive studies have been carried out to synthesize acridine and its derivatives utilizing different catalytic agents, such as: glycerol [14], $BF_3.Et_2O$ [15], Ceric ammonium nitrate [16] and Amberlyst-15 [1]. In previous work, we also reported the synthesis of acridine and its derivatives utilizing ionic-liquids as acid catalyst [17, 18].

Whilst the syntheses of organic compounds catalyzed by several transition metal complexes have been reported, reactions catalyzed by Schiff-base metal complexes have received less attention. The salen-type ligand is one of well-known Schiff-base ligand that its metal complexes have widely been employed widely as catalyst of more organic reactions [19, 20], fluorescent sensor [21], photo degradation of organic dyes [22], antimicrobial activities [23], it is also used in DNA cleavage [24] and many other catalytic purposes. Schiff-base metal complexes are also considered as ideal "green" catalysts due to their low toxicities. Easy preparations and inexpensive starting materials are other

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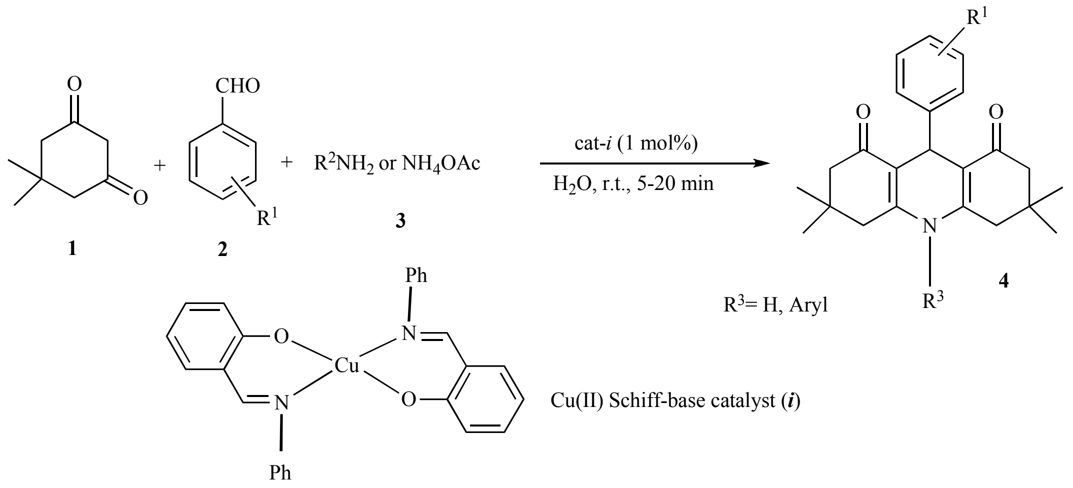

Scheme 1. Synthesis of 1,8-dioxodecahydroacridines in the presence of Cu(II) Schiff-base catalyst (*i*).

advantage of Schiff-base metal complexes. In this work, we report an efficient, high yield and rapid method for synthesis of acridine and derivatives by new reusable Cu(II) Schiff-base catalyst (*i*) [25].

A three-component condensation of dimedone **1** (2 mmol), 4-chlorobenzaldehyde **2** (1 mmol) and *p*-toluidine **3** (1 mmol) was employed to optimize the reaction conditions with respect to temperature, reaction time, solvent, molar ratio of catalyst to the substrate and the reusability of the catalyst. The reaction was carried out under aqueous conditions at room temperature and it was found that 1 mol% of Cu(II) Schiff-base catalyst was sufficient to obtain the desired 1,8-dioxodecahydroacridines in 97% yield within 7 min (Scheme **1**).

EXPERIMENTAL

Materials and Methods: NMR spectra were determined on a Fourier-transform (FT)- NMR Bruker AV-400 spectrometer in DMSO-d_6 and CDCl$_3$ are expressed in δ values relative to tetramethylsilane; coupling constants (J) are measured in Hz. Melting points were determined on a ELECTR THERMAL9100. Infrared spectra were recorded on a RAYLEIGH WQF-510 Fourier transform instrument. Commercially available reagents were used throughout without further purification.

General procedure for the synthesis of 1,8-dioxodecahydroacridine derivatives: A mixture of dimedone (2.0 mmol), aromatic aldehyde (1.0 mmol), aromatic amine or ammonium acetate (1.0 mmol) and Cu(II) Schiff-base catalyst (1 mol%) in water (2 mL) was stirred at room temperature for an appropriate time (5-20 min). The progress of the reaction was monitored by TLC (*n*-hexan/ethyl acetate 4:1). After completion of the reaction, the resulting solid crude product was filtered and then recrystallized from ethanol–water to obtain pure product. The physical data (M.p, IR, NMR) of these known compounds were found to be identical with those reported in the literature.

3,3,6,6-tetramethyl-9-(4-nitrophenyl)-10-phenyl-3,4,6,7,9,10-hexahydroacridine-(2H,5H)-dione (Table 3, entry 12): Yield: 98%; M.p 257-259 °C; IR spectrum (KBr, ν, cm^{-1}): 785 (–CH out of bending of aromatic ring), 1235 (CN stretching), 1345, 1551 (NO$_2$ stretching), 1375, 1575 (C=C– stretching of aromatic ring), 1671 (C=O– of 1,3-diketone), 2945 (–CH stretching of aliphatic), 3055 (–CH stretching of aromatic ring); ^{1}H NMR spectrum (400 MHz, CDCl$_3$, δ, ppm): 1.63 (s, 6H, 2 * CH$_3$), 1.81 (s, 6H, 2 * CH$_3$), 2.51 (dd, 4H, J= 16.2 Hz, 2 * CH$_2$), 2.71 (dd, 4H, J= 16.3 Hz, 2 * CH$_2$), 5.07 (s, 1H, CH), 7.21-7.34 (m, 5H, ArH), 7.51 (d, 2H, J= 9.5 Hz, ArH), 7.63 (d, J= 9.3 Hz, 2H, ArH).

9-(2-chlorophenyl)-10-(4-chlorophenyl)-3,3,6,6-tetramethyl-3,4,6,7,9,10-hexahydroacridine-1,8(2H,5H)-dione (Table 3, entry 24): Yield: 95%; M.p 319-321 °C; IR spectrum (KBr, ν, cm^{-1}): 978 (–CH out of bending of aromatic ring), 1237 (CN stretching), 1365, 1570 (C=C– stretching of aromatic ring), 1675 (C=O– of 1,3-diketone), 2957 (–CH stretching of aliphatic), 3057 (–CH stretching of aromatic ring); ^{1}H NMR spectrum (400 MHz, CDCl$_3$, δ, ppm): 1.71 (s, 6H, 2 * CH$_3$), 1.79 (s, 6H, 2 * CH$_3$), 2.33 (dd, 4H, J= 16.1 Hz, 2 * CH$_2$), 2.40 (dd, 4H, J= 16.4 Hz, 2 * CH$_2$), 5.24 (s, 1H, CH), 7.31 (d, 2H, J= 9.3 Hz, ArH), 7.35 (d, 1H, J= 7.8 Hz, ArH), 7.38-7.43 (m, 2H, ArH), 7.47 (d, 2H, J= 9.2 Hz, ArH), 7.51 (d, 1H, J= 7.7 Hz, ArH).

9,10-bis(4-chlorophenyl)-3,3,6,6-tetramethyl-3,4,6,7,9,10-hexahydroacridine-(2H,5H)-dione (Table 3, entry 25): Yield: 96%; M.p 305-307 °C; IR spectrum (KBr, ν, cm^{-1}): 778 (–CH out of bending of aromatic ring), 1225 (CN stretching), 1367, 1545 (C=C– stretching of aromatic ring), 1671 (C=O– of 1,3-diketone), 2953 (–CH stretching of aliphatic), 3065 (–CH stretching of aromatic ring); ^{1}H NMR spectrum (400 MHz, CDCl$_3$, δ, ppm): 1.71 (s, 6H, 2 * CH$_3$), 1.76 (s, 6H, 2 * CH$_3$), 2.35 (dd, 4H, J= 16.1 Hz, 2 * CH$_2$), 2.43 (dd, 4H, J= 16.3 Hz, 2 * CH$_2$), 5.31 (s, 1H, CH), 7.21 (d, 2H, J= 9.4 Hz, ArH), 7.32 (d, 2H, J= 9.4 Hz, ArH), 7.47 (d, 2H, J= 9.3 Hz, ArH), 7.52 (d, 2H, J= 9.1 Hz, ArH).

RESULTS AND DISCUSSION

To investigate the solvent effect the Cu(II) Schiff-base catalyst (*i*) was tested as a model reaction between dimedone, *p*-toluidine and 4-chlorobenzaldehyde in different reaction media. The results are gathered in Table **1**. The obtained results show that the reaction operates better in polar solvents than non-polar ones. Among the polar

Table 1. **Solvent Effect on the Reaction Between Dimedone, *p*-Toluidine and 4-Chlorobenzaldehyde**

Solvent	H_2O	C_2H_5OH	CH_3CN	THF	Toluene
Time (min)	7	7	10	15	19
Yield (%)	97	96	92	89	85

Reaction condition: [a] 4-chlorobenzaldehyde (1 mmol), dimedone (2 mmol) *p*-toluidine (1 mmol), Cu(II) Schiff-base catalyst (1 mol %), solvent (2 mL); [b] Isolated yield.

solvents examined, water was found to be the most effective solvent. Remarkably, the condensation reaction preceded smoothly both in water and ethanol conditions to afford desired product in good yields. This result confirms that the Cu(II) Schiff-base catalyst (*i*) is not only a water-stable catalyst but also demonstrates high catalytic activity.

To underline the merit of this method, the one-pot synthesis of 1,8-dioxodecahydroacridines using Cu(II) Schiff-base catalyst (*i*) was faster and gave higher yields than those catalysts reported in the literature (Table **2**). According to the results, the catalyst amounts for the most systems are greater than 1 mol% which requires longer reaction times and lower yields than this catalytic system.

To study the generality of this process, the reaction was carried out with a variety of aldehyde derivatives and aromatic amines to synthesize the corresponding polyfunctionalized -dioxo-9,10-aryl-decahydroacridines. The results are summarized in Table **3**. In this way, a series of aromatic aldehydes and amines underwent electrophilic substitution reaction with dimedone to afford a wide range of

Table 2. **Reaction Between Dimedone, 4-Chlorobenzaldehyde and *p*-Toluidine in Presence of Different Catalysts**

Entry	Catalyst	Catalyst (mol%)	Time (min)	Yield (%)	Ref.
1	Cu(II) Schiff-base	1	7	97	This work
2	$C_{11}H_{15}COOH$	10	360	26.8	[26a]
3	PTSA	2	360	18	[12]
4	$Sc(DS)_3$	10	360	78.3	[26a]
5	[HMIM]TFA	0.1 gr	300	84	[26b]
6	$C_7H_{15}COOH$	2	360	31	[12]
7	TsOH	10	360	13.2	[26a]
8	DBSA	2	360	41	[12]

Table 3. **Synthesis of 1,8-Dioxodecahydroacridine Derivatives in the Presence of Cu(II) Schiff-Base Catalyst**

Entry	R^1	R^2	Mp (°C)	Time (min)	Yield (%) [Ref.]
1	4-H	NH_4OAc	281-283	20	93 [2]
2	4-Cl	NH_4OAc	295-297	14	95 [2]
3	4-OH	NH_4OAc	303-305	17	93 [2]
4	4-OMe	NH_4OAc	269-271	15	93 [27a]
5	4-Me	NH_4OAc	320-322	17	95 [27b]
6	3-NO_2	NH_4OAc	285-287	14	96 [2]
7	4-NO_2	NH_4OAc	315-317	11	98 [27c]
8	4-H	Aniline	255-257	13	94 [26b]
9	4-Cl	Aniline	245-247	10	95 [27d]
10	4-OMe	Aniline	217-219	10	95 [26b]
11	3-NO_2	Aniline	295-297	8	97 [16]
12	4-NO_2	Aniline	257-259	7	98
13	4-H	*p*-toluidine	263-265	12	94 [27e]
14	2-Cl	*p*-toluidine	287-289	9	95 [26a]
15	4-Cl	*p*-toluidine	273-275	7	97 [27e]
16	4-OH	*p*-toluidine	347-349	10	93 [26a]
17	4-OMe	*p*-toluidine	281-283	10	94 [27e]
18	4-Me	*p*-toluidine	295-297	8	96 [27e]
19	3-NO_2	*p*-toluidine	283-285	5	98 [27e]
20	4-H	4-methoxyaniline	217-219	14	95 [12]
21	4-Cl	4-methoxyaniline	251-253	9	97 [12]
22	4-OMe	4-methoxyaniline	208-210	9	95 [12]
23	4-Me	4-methoxyaniline	238-240	10	96 [12]
24	2-Cl	4-chloroaniline	319-321	11	95
25	4-Cl	4-chloroaniline	305-307	8	96

Table 4. Reusability Studies of the Catalyst for Synthesis of 9-(4-Chlorophenyl)-3,3,6,6-Tetramethyl-10-*p*-tolyl-3,4,6,7,9,10-Hexahydroacridine-(2*H*,5*H*)-dione (Table 4- Entry 15)

Number of Experiments	Fresh	1	2	3	4
Isolated yield (%)	97	96	95	95	93

substituted 1,8-dioxodecahydroacridines in good to excellent yields. As shown in Table **3**, the electronic property of the substituents attached on the aromatic ring has an effective role in the conversion rate. So that, aromatic aldehydes with electron-withdrawing groups on the aromatic ring (Table **3**, entries 6, 7, 11, 12, 18) react faster than electron-donating groups (Table **3**, entries 3, 4, 5, 10, 17, 21, 25). Furthermore, both aromatic amines and ammonium acetate similarly underwent well to the conversion.

The reusability of the catalysts was checked using dimedone, 4-chlorobenzaldehyde and *p*-toluidine as a model substrate. In this regard, at the end of the reaction, ethyl acetate was added to the reaction mixture. The aqueous layer was then separated and used directly in the next reaction without further purification. As shown in (Table **4**), the recovered catalyst was reused at five successive runs without appreciable loss in its catalytic activity.

CONCLUSION

In summary, Cu(II) Schiff-base was used as an efficient catalyst for the synthesis of 1,8-dioxodecahydroacridines which resulted in better yields. Cu(II) Schiff-base effective catalysis the one-pot three-component condensation of dimedone, aromatic aldehydes and aromatic amines or ammonium acetate in water to produce 1,8-dioxodecahydroacridines in excellent yields. The catalyst offers several advantages including shorter reaction times, high yield of the products, mild reaction conditions, lower catalytic loading, cleaner reactions, green solid acid catalyst as well as simple experimental and isolation procedures. Also, the catalysts were able to be reused easily for five-run test with a smooth decrease in the catalytic activity of the recovered catalyst.

CONFLICT OF INTEREST

The authors confirm that this article content has no conflict of interest.

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