

Research Article

Poly(4-Vinylpyridinium)Hydrogen Sulfate Catalyzed an Efficient and Ecofriendly Protocol for the One-Pot Multicomponent Synthesis of 1,8-Acridinediones in Aqueous Medium

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Highly efficient, ecofriendly, and improved protocol for the synthesis of 1,8-acridinediones has been developed *via* one-pot multicomponent condensation of 1,3-cyclohexanedione/dimedone, aromatic aldehydes, and ammonium acetate utilizing poly(4-vinylpyridinium)hydrogen sulfate as catalyst in aqueous medium. Excellent yields in shorter reaction time, simple work-up procedure, easy recovery, and reusability of the catalyst are attractive features of this green protocol.

1. Introduction

Multicomponent reactions (MCRs) have emerged as an efficient and powerful tool in modern organic chemistry towards the generation of highly diverse and complex product from readily available substrates in a single operation without isolation of intermediates in minimal time with maximum selectivity, high atom economy, and high purity with excellent yields [1]. In addition, reactions under aqueous medium have received considerable attention in organic synthesis, because of both economic and environmental safety reasons [2]. Moreover, heterogeneous catalysts have advantages over homogeneous catalysts in terms of efficiency, operational simplicity, recovery from the reaction mixture, and reusability [3].

1,8-Acridinediones having 1,4-dihydropyridine (1,4-DHP) parent nucleus, which are versatile intermediates in the synthesis of numerous pharmaceuticals including those for the treatment of cardiovascular diseases and hypertension as effective calcium channel blockers [4, 5], Alzheimer's disease, and as chemosensitizer in tumor therapy

[6, 7], are also used as laser dyes [8, 9] and photo initiators for radical polymerization reactions [10–12].

In view of the above applications, many methods have been developed including microwave irradiation [13, 14], using various catalytic systems such as *p*-toluenesulfonic acid [15], ceric ammonium nitrate [16], InCl_3 [17], $\text{CeCl}_3 \cdot 7\text{H}_2\text{O}$ [18], L-proline, $\text{Zn}(\text{OAc})_2 \cdot 7\text{H}_2\text{O}$ [19], $\text{In}(\text{OTf})_3$ [20], MCM-41- SO_3H [21], Amberlite IR-120H [22], methanesulfonic acid [23], silica bonded *s*-sulfonic acid [24], silica-supported preysler nanoparticles [25], *p*-dodecylbenzenesulfonic acid [26], carbon based solid acid [27], and ionic liquids [28, 29]. However, most of these methods suffer from drawbacks such as low yield of the product, longer reaction time, multistep process, formation of side products, critical isolation procedure, use of hazardous organic solvents and expensive catalysts, difficult in recovery and reusability of the catalyst. Thus, there is a need for the development of new and efficient methods for the preparation of such compounds in high yields and under mild reaction conditions.

In continuation of our studies towards the development of novel methodologies for the synthesis of biologically



A plausible mechanism for the formation of 1,8-acridinediones catalysed by P(4-VPH)HSO₄ is shown in Scheme 2. In the presence of catalyst, the electrophilicity of aldehydic carbonyl carbon increases and readily reacts with the enolic form of 1,3-dicarbonyl compounds which resulted the formation of chalcone derivative [C]. Compound [C] reacts with aminated 1,3-dicarbonyl compound [B] which is formed *in situ* by the reaction of 1,3-dicarbonyl compound with ammonium acetate that furnished the corresponding 1,8-acridinediones through the cyclisation followed by the dehydration.

TABLE 2: P(4-VPH)HSO₄ catalyzed one-pot synthesis of 1,8-acridinediones (**5a–k** and **6a–k**).

Entry ^a	Aldehyde	Product	Time (min)	Yield ^b (%)	Melting point (°C)	
					Found	Lit. [Ref.]
1	Benzaldehyde	5a	15	92	278–280	279–281 [16]
2	4-Chlorobenzaldehyde	5b	10	94	297–299	298–299 [16]
3	3-Nitrobenzaldehyde	5c	15	89	278–281	282–284 [16]
4	4-Methoxybenzaldehyde	5d	10	91	306–308	304–306 [16]
5	3,4-Dimethoxybenzaldehyde	5e	10	93	355–356	—
6	4-Hydroxybenzaldehyde	5f	15	90	305–307	307–309 [16]
7	3-Methoxy-4-hydroxybenzaldehyde	5g	15	90	281–283	—
8	3-Ethoxy-4-hydroxybenzaldehyde	5h	20	88	299–302	—
9	5-Bromo-2-hydroxybenzaldehyde	5i	20	91	272–274	—
10	3,5-Dibromo-2-hydroxybenzaldehyde	5j	20	90	250–251	—
11	5-Chloro-2-hydroxybenzaldehyde	5k	20	92	295–297	—
12	Benzaldehyde	6a	10	93	258–260	258–260 [18]
13	4-Chlorobenzaldehyde	6b	10	95 (93, 90, 89, 86, 84) ^c	299–301	298–300 [18]
14	3-Nitrobenzaldehyde	6c	15	88	290–293	294–296 [18]
15	4-Methoxybenzaldehyde	6d	10	90	273–274	272–273 [23]
16	3,4-Dimethoxybenzaldehyde	6e	10	94	259–261	258–260 [13]
17	4-Hydroxybenzaldehyde	6f	15	92	361–363	>300 [23]
18	3-Methoxy-4-hydroxybenzaldehyde	6g	15	92	294–296	294–295 [18]
19	3-Ethoxy-4-hydroxybenzaldehyde	6h	15	89	280–282	—
20	5-Bromo-2-hydroxybenzaldehyde	6i	20	90	254–255	—
21	3,5-Dibromo-2-hydroxybenzaldehyde	6j	20	90	294–295	—
22	5-Chloro-2-hydroxybenzaldehyde	6k	20	92	234–236	—

^a Reaction conditions: 1,3-cyclohexanedione/dimedone (2 mmol), arylaldehyde (1 mmol), ammonium acetate (3 mmol), and P(4-VPH)HSO₄ (0.02 g), heat at 80°C for 10–20 min in 5 mL of H₂O.

^b Isolated yields.

^c Yields refer to the reusability of the catalyst over additional five times.

TABLE 3: Comparing the catalytic efficiency of P(4-VPH)HSO₄ for the synthesis of **6b** with some reported catalysts.

Entry ^a	Catalyst (g)	Reaction conditions	Time (min)	Yield (%) ^{Ref.}
1	L-Proline (0.041 g)	H ₂ O, Reflux	120	88 [19]
2	Zn(OAc)·2H ₂ O (0.0172 g)	H ₂ O, Reflux	120	91 [19]
3	CAN (0.0274 g)	PEG 400, Rt	25	92 [16]
4	InCl ₃ (0.0022 g)	EtOH, Rt	27	93 [17]
5	CeCl ₃ ·7H ₂ O (0.0182 g)	Ionic liquid, 100°C	180	89 [18]
6	MSA ^a (0.0096 g)	Solvent-free, 120°C	60	92 [23]
7	CBSA ^b (0.0056 g)	Solvent-free, 100°C	15	90 [27]
8	P(4-VPH)HSO ₄ (0.02 g)	H ₂ O, 80°C	10	95 [present work]

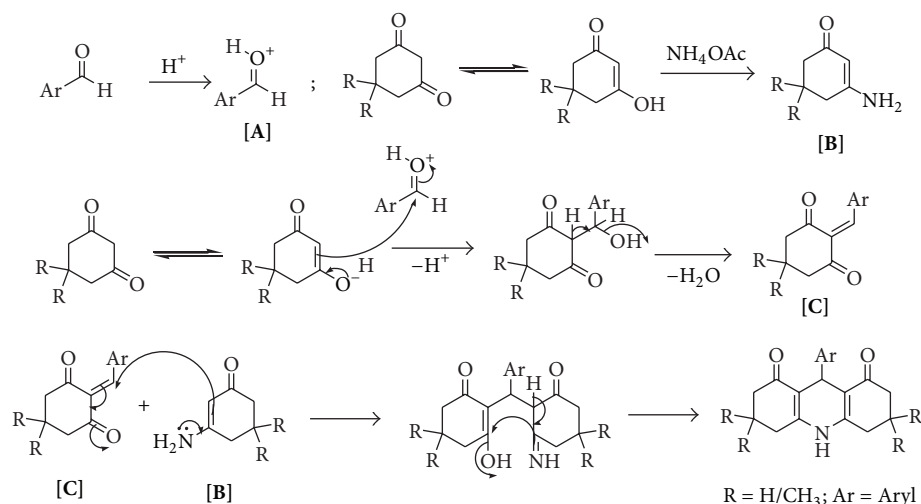
^a MSA: methanesulfonic acid; ^b CBSA: carbon-based solid acid

For comparison, mole percentages were converted into grams.

3. Experimental

All the chemicals were procured from Aldrich/Merck, and solvents were used without further purification. Melting points were recorded on Stuart SMP30 apparatus and are uncorrected. Thin layer chromatography was performed with F254 silica-gel precoated sheets using hexane/ethyl acetate (8:2) as eluent, visualized by UV light and iodine

vapor. Products were characterized by comparing with the authentic samples and by spectral data (IR, ¹H NMR, and Mass). IR spectra were recorded on Perkin-Elmer 100S spectrophotometer using KBr disk. ¹H NMR spectra were recorded on Bruker 400 MHz spectrometer using DMSO-*d*₆ as solvent and TMS as internal standard. Elemental analysis was performed on a Carlo Erba model EA1108. Mass spectra were recorded on a Jeol JMSD-300 spectrometer.



SCHEME 2: Proposed mechanism for the formation of 1,8-acridinediones.

3.1. General Procedure for the Synthesis of 1,8-Acridinediones. To a mixture of 1,3-cyclohexanedione/dimedone (2 mmol), arylaldehyde (1 mmol), and ammonium acetate (3 mmol) in 5 mL of water, 0.02 g of poly(4-vinylpyridinium)hydrogen sulfate was added and stirred at 80°C for an appropriate time as shown in Table 2. After completion of the reaction (TLC monitoring), the solid separated out was filtered, washed with water, dried, and recrystallized from ethanol/acetic acid to afford the pure 1,8-acridinediones in excellent yields. Aqueous layer containing catalyst was recovered under reduced pressure, dried, and reused for additional five times for subsequent reactions.

3.2. Spectral Data of New Compounds

3.2.1. 9-(3,4-Dimethoxyphenyl)-3,4,6,7,9,10-hexahydro-2H,5H-acridine-1,8-dione (5e). Pale yellow solid; IR (KBr) ν_{\max} (cm⁻¹): 3417 (NH), 1635 (C=O), 1597 (C=C), 1026 (C–O–C); ¹H NMR (400 MHz, DMSO-*d*₆): δ 1.75–1.79 (m, 2H), 1.83–1.90 (m, 2H), 2.16–2.19 (m, 4H), 2.45–2.48 (m, 4H), 3.47 (s, 3H), 3.49 (s, 3H), 4.83 (s, 1H), 6.47–6.49 (m, 1H), 6.62 (d, *J* = 8.0 Hz, 1H), 6.74 (s, 1H), 9.42 (s, 1H); MS (ESI) *m/z*: 354 (M + H)⁺; anal. calcd. for C₂₁H₂₃NO₄: C, 71.37; H, 6.56; N, 3.96; found: C, 71.09; H, 6.71; N, 3.76.

3.2.2. 9-(4-Hydroxy-3-methoxyphenyl)-3,4,6,7,9,10-hexahydro-2H,5H-acridine-1,8-dione (5g). Pale yellow solid; IR (KBr) ν_{\max} (cm⁻¹): 3411 (NH), 3302 (OH), 1637 (C=O), 1596 (C=C), 1132 (C–O–C); ¹H NMR (400 MHz, DMSO-*d*₆): δ 1.76–1.80 (m, 2H), 1.86–1.91 (m, 2H), 2.17–2.20 (m, 4H), 2.45–2.49 (m, 4H), 3.47 (s, 3H), 4.80 (s, 1H), 6.46–6.47 (m, 1H), 6.55 (d, *J* = 8.0 Hz, 1H), 6.73 (s, 1H), 8.49 (s, 1H), 9.41 (s, 1H); MS (ESI) *m/z*: 340 (M + H)⁺; anal. calcd. for C₂₀H₂₁NO₄: C, 70.78; H, 6.24; N, 4.13; found: C, 70.54; H, 6.43; N, 4.22.

3.2.3. 9-(3-Ethoxy-4-hydroxyphenyl)-3,4,6,7,9,10-hexahydro-2H,5H-acridine-1,8-dione (5h). Red solid; IR (KBr) ν_{\max}

(cm⁻¹): 3414 (NH), 3329 (OH), 1644 (C=O), 1591 (C=C), 1119 (C–O–C); ¹H NMR (400 MHz, DMSO-*d*₆): δ 1.29 (t, *J* = 7.2 Hz, 3H), 1.77–1.81 (m, 2H), 1.88–1.93 (m, 2H), 2.19–2.22 (m, 4H), 2.47–2.49 (m, 4H), 3.88–3.93 (m, 2H), 4.81 (s, 1H), 6.45–6.47 (m, 1H), 6.55 (d, *J* = 8.0 Hz, 1H), 6.71 (s, 1H), 8.48 (s, 1H), 9.36 (s, 1H); MS (ESI) *m/z*: 354 (M + H)⁺; anal. calcd. for C₂₁H₂₃NO₄: C, 71.37; H, 6.56; N, 3.96; found: C, 71.18; H, 6.69; N, 3.80.

3.2.4. 9-(5-Bromo-2-hydroxyphenyl)-3,4,6,7,9,10-hexahydro-2H,5H-acridine-1,8-dione (5i). Pale yellow solid; IR (KBr) ν_{\max} (cm⁻¹): 3414 (NH), 3279 (OH), 1625 (C=O), 1593 (C=C), 615 (C–Br); ¹H NMR (400 MHz, DMSO-*d*₆): δ 1.78–1.83 (m, 2H), 1.92–1.96 (m, 2H), 2.26–2.30 (m, 4H), 2.55–2.61 (m, 4H), 4.84 (s, 1H), 6.65 (d, *J* = 8.4 Hz, 1H), 6.87 (d, *J* = 8.4 Hz, 1H), 7.09–7.12 (m, 1H), 9.82 (s, 1H), 9.91 (s, 1H); MS (ESI) *m/z*: 411 (M + Na)⁺; anal. calcd. for C₁₉H₁₈BrNO₃: C, 58.78; H, 4.67; N, 3.61; found: C, 58.62; H, 4.83; N, 3.79.

3.2.5. 9-(3,5-Dibromo-2-hydroxyphenyl)-3,4,6,7,9,10-hexahydro-2H,5H-acridine-1,8-dione (5j). Yellow solid; IR (KBr) ν_{\max} (cm⁻¹): 3415 (NH), 3285 (OH), 1620 (C=O), 1580 (C=C), 616 (C–Br); ¹H NMR (400 MHz, DMSO-*d*₆): δ 1.83–1.91 (m, 2H), 1.93–1.97 (m, 2H), 2.30–2.33 (m, 4H), 2.57–2.64 (m, 4H), 4.85 (s, 1H), 6.83 (s, 1H), 7.49 (s, 1H), 9.91 (s, 1H), 10.72 (s, 1H); MS (ESI) *m/z*: 468 (M + H)⁺; anal. calcd. for C₁₉H₁₇Br₂NO₃: C, 48.85; H, 3.67; N, 3.00; found: C, 48.64; H, 3.86; N, 3.12.

3.2.6. 9-(5-Chloro-2-hydroxyphenyl)-3,4,6,7,9,10-hexahydro-2H,5H-acridine-1,8-dione (5k). Pale yellow solid; IR (KBr) ν_{\max} (cm⁻¹): 3414 (NH), 3280 (OH), 1622 (C=O), 1593 (C=C), 617 (C–Cl); ¹H NMR (400 MHz, DMSO-*d*₆): δ 1.79–1.82 (m, 2H), 1.92–1.96 (m, 2H), 2.26–2.29 (m, 4H), 2.54–2.60 (m, 4H), 4.84 (s, 1H), 6.70 (d, *J* = 8.4 Hz, 1H), 6.74 (d, *J* = 8.4 Hz, 1H), 6.97–7.00 (m, 1H), 9.84 (s, 1H), 9.89 (s, 1H); MS (ESI) *m/z*: 344 (M + H)⁺; anal. calcd. for C₁₉H₁₈ClNO₃: C, 66.38; H, 5.28; N, 4.07; found: C, 66.17; H, 5.40; N, 4.26.

3.2.7. 9-(3-Ethoxy-4-hydroxyphenyl)-3,3,6,6-tetramethyl-3,4,6,7,9,10-hexahydro-2H,5H-acridine-1,8-dione (**6h**). Pale yellow solid; IR (KBr) ν_{\max} (cm^{-1}): 3416 (NH), 3277 (OH), 1620 (C=O), 1598 (C=C), 1122 (C–O–C); ^1H NMR (400 MHz, DMSO- d_6): δ 0.88 (s, 6H), 1.01 (s, 6H), 1.28 (t, J = 7.2 Hz, 3H), 1.99 (d, J = 16.0 Hz, 2H), 2.17 (d, J = 16.0 Hz, 2H), 2.30 (d, J = 16.8 Hz, 2H), 2.43 (d, J = 16.8 Hz, 2H), 3.85–3.91 (m, 2H), 4.70 (s, 1H), 6.49–6.56 (m, 2H), 6.68 (s, 1H), 8.48 (s, 1H), 9.20 (s, 1H); MS (ESI) m/z : 432 ($M + \text{Na}$) $^+$; anal. calcd. for $\text{C}_{25}\text{H}_{31}\text{NO}_4$: C, 73.32; H, 7.63; N, 3.42; found: C, 73.18; H, 7.77; N, 3.56.

3.2.8. 9-(5-Bromo-2-hydroxyphenyl)-3,3,6,6-tetramethyl-3,4,6,7,9,10-hexahydro-2H,5H-acridine-1,8-dione (**6i**). White solid; IR (KBr) ν_{\max} (cm^{-1}): 3415 (NH), 3279 (OH), 1622 (C=O), 1570 (C=C), 656 (C–Br); ^1H NMR (400 MHz, DMSO- d_6): δ 0.89 (s, 6H), 0.96 (s, 3H), 1.04 (s, 3H), 2.03 (d, J = 16.0 Hz, 2H), 2.22–2.57 (m, 6H), 5.03 (s, 1H), 6.95 (d, J = 8.8 Hz, 1H), 7.03 (s, 1H), 7.26–7.29 (m, 1H), 9.86 (s, 1H), 10.60 (s, 1H); MS (ESI) m/z : 445 ($M + \text{H}$) $^+$; anal. calcd. for $\text{C}_{23}\text{H}_{26}\text{BrNO}_3$: C, 62.17; H, 5.90; N, 3.15; found: C, 62.04; H, 5.99; N, 3.31.

3.2.9. 9-(3,5-Dibromo-2-hydroxyphenyl)-3,3,6,6-tetramethyl-3,4,6,7,9,10-hexahydro-2H,5H-acridine-1,8-dione (**6j**). Pale yellow solid; IR (KBr) ν_{\max} (cm^{-1}): 3416 (NH), 3284 (OH), 1635 (C=O), 1613 (C=C), 665 (C–Br); ^1H NMR (400 MHz, DMSO- d_6): δ 0.93 (s, 6H), 1.04 (s, 6H), 2.13 (d, J = 16.4 Hz, 2H), 2.30 (d, J = 16.4 Hz, 2H), 2.46 (d, J = 17.6 Hz, 4H), 4.82 (s, 1H), 6.88 (s, 1H), 7.49 (s, 1H), 9.87 (s, 1H), 10.63 (s, 1H); MS (ESI) m/z : 524 ($M + \text{H}$) $^+$; anal. calcd. for $\text{C}_{23}\text{H}_{25}\text{Br}_2\text{NO}_3$: C, 52.79; H, 4.82; N, 2.68; found: C, 52.65; H, 4.97; N, 2.80.

3.2.10. 9-(5-Chloro-2-hydroxyphenyl)-3,3,6,6-tetramethyl-3,4,6,7,9,10-hexahydro-2H,5H-acridine-1,8-dione (**6k**). White solid; IR (KBr) ν_{\max} (cm^{-1}): 3415 (NH), 3285 (OH), 1629 (C=O), 1579 (C=C), 656 (C–Cl); ^1H NMR (400 MHz, DMSO- d_6): δ 0.89 (s, 6H), 0.97 (s, 3H), 1.04 (s, 3H), 2.03 (d, J = 16.0 Hz, 2H), 2.24 (d, J = 16.0 Hz, 2H), 2.33 (d, J = 17.2 Hz, 2H), 2.54 (d, J = 17.2 Hz, 2H), 5.03 (s, 1H), 6.90 (s, 1H), 7.00 (d, J = 8.8 Hz, 1H), 7.14–7.17 (m, 1H), 9.82 (s, 1H), 10.58 (s, 1H); MS (ESI) m/z : 400 ($M + \text{H}$) $^+$; anal. calcd. for $\text{C}_{23}\text{H}_{26}\text{ClNO}_3$: C, 69.08; H, 6.55; N, 3.50; found: C, 68.92; H, 6.73; N, 3.68.

4. Conclusion

In conclusion, we have developed a simple, mild, and efficient protocol for the synthesis of 1,8-acridinediones utilizing poly(4-vinylpyridinium)hydrogen sulphate as catalyst in aqueous medium. Excellent yields in shorter reaction times, easy work-up procedure, environmentally benign nature, recovery, and reusability of the catalyst are the obvious advantages of this methodology.

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