

Short communication

An eco-friendly procedure for the synthesis of polysubstituted quinolines under aqueous media

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Abstract

Some new metal dodecyl sulfates were prepared by the reaction of sodium dodecyl sulfate and the corresponding metal nitrates. All of the prepared salts catalyzed the synthesis of quinoline from *o*-aminoaryl ketones and ketones or β -diketones. Among the metal dodecyl sulfates, zirconium tetrakis(dodecyl) sulfate, was superior to others. Polysubstituted quinolines were synthesized from the same starting materials in the presence of a catalytic amount of $Zr(DS)_4$ in good to high yields in water under reflux conditions. $Zr(DS)_4$ could be recovered and reused. © 2006 Elsevier B.V. All rights reserved.

Keywords: β -Diketones; *o*-Aminoaryl ketones; Quinoline; Dodecyl sulfate salts; Zirconium tetrakis(dodecyl)sulfate; Metal nitrate; Water

Organic reactions in water are now of great interest because water is a cheap, safe and clean solvent [1]. Industry prefers to use water as a solvent rather than toxic organic solvents [2]. Although water is inexpensive, non-toxic and non-flammable, but it is not routinely used as a solvent for organic reactions. Susceptibility of water for reaction with most Lewis acids and intermediates and insolubility of most organic compounds in water, are the major problems for applying it as a unique solvent in chemical processes. For overcoming of these limitations, the synthesis and applications of rare-earth metal triflates as efficient water active Lewis acids for running organic reactions in aqueous media were developed [3]. The above mentioned catalysts have their own drawbacks, such as expensive and long reaction times or in some cases they need organic co-solvents.

It is well-known that the quinoline skeleton plays an important role as an intermediate for the design of antitumor, anti-malarial, antiviral, antibacterial and anti-inflammatory agents [4]. The Friedlander annulation, among the other methods, is one of the most simple and straightforward approaches for the synthesis of quinoline derivatives. Varieties of catalysts have

been reported for this purpose. However, almost all of them were used in organic solvents [5]. Very recently, synthesis of quinoline derivatives by the Friedlander method was reported in water but in the presence of stoichiometric amounts of hydrochloric acid and in 60–90 °C [6].

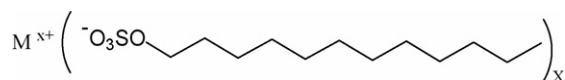
In continuation of our studies on the synthesis of quinolines [7], 1,4-dihydropyridines [8] and pyridines [9], we were interested to design some new, cheap and powerful Lewis acids for Friedlander method. Synthesis, characterization and applications of scandium tris(dodecyl sulfate) [10] and aluminum tris(dodecyl sulfate) [11] has already been reported. The successful applications of these Lewis acids led us to prepare some combined Lewis acid-surfactant catalysts (LASC) and to investigate their applications in organic functional group transformations under aqueous media.

Herein, we wish to report a group of surfactants, which are highly effective and water-insensitive species that facilitate the Friedlander quinoline synthesis in water.

Metal dodecyl sulfates (Scheme 1) were synthesized by addition a solution of sodium dodecyl sulfate to an aqueous solution of metal nitrates. The products were precipitated immediately. The results are summarized in Table 1. As could be seen, Fe^{3+} , Al^{3+} , Cr^{3+} , Ca^{2+} , Sr^{2+} , Pb^{2+} and Zr^{4+} dodecyl sulfates were prepared at room temperature in good yields. Ce^{4+} salt was obtained

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Scheme 1. The structure of metal dodecyl sulfates.

in moderate yield at ambient temperature. Both Ag^+ and Co^{2+} dodecyl sulfates were produced in lower yields after longer periods under reflux conditions (Table 1, entries 4 and 14).

Other nitrates, such as Cd^{2+} , Mn^{2+} , Mg^{2+} , Zn^{2+} and Cu^{2+} were not reacted with sodium dodecyl sulfate even after 12 h under reflux conditions (Table 1). Then, catalytic activity of synthesized metal dodecyl sulfates was studied by synthesis of **3b** from 2-amino-5-chlorobenzophenone and ethyl acetoacetate as a model reaction in $\text{EtOH}:\text{H}_2\text{O}$ (1:2) as solvent under reflux conditions. Although the obtained results showed that the salts catalyzed this reaction with moderate to excellent yields (Table 2), but $\text{Zr}(\text{DS})_4$ gave the best result. The results also

showed that water itself was the best solvent (Table 3, entry 4). $\text{Zr}(\text{DS})_4$ was recovered through separation by a centrifuge and reused as catalyst for a new run (Table 2, entry 8). Therefore, we applied these conditions for the synthesis of others quinolines.

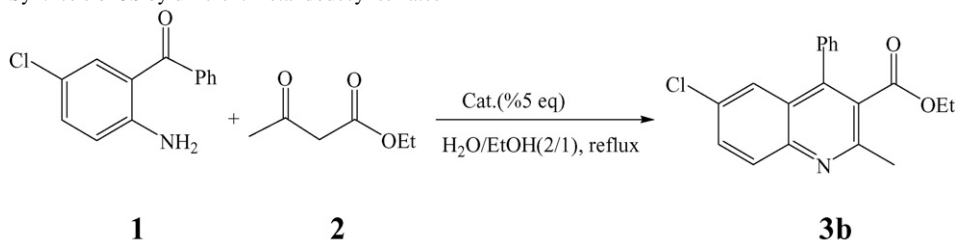
A good range of *o*-aminoaryl ketones and β -diketones were condensed to the corresponding quinoline derivatives in the presence of catalytic amounts of $\text{Zr}(\text{DS})_4$ in good to high yields in water under reflux conditions. Surprisingly, reactions of cyclic β -dicarbonyl compounds were faster than others and products obtained in excellent yields (Table 3, entries 1, 4, 7 and 8). An interesting example is the synthesis of a 4-hydroxy quinoline derivative from the reaction of 2-aminobenzonitrile with dimedone (Table 3, entry 13). We also found that the reactions of simple ketones, such as cyclohexanone, with *o*-aminoaryl ketones progressed smoothly in high yields.

We believe that this new type of “combined Lewis acid-surfactant catalyst”, would act in dual role both as a catalyst to

Table 1
Preparation of metal dodecyl sulfates

Entry	Metal nitrate	Salt	Temp. ($^{\circ}\text{C}$)	Time (h)	Yield (%)	Color of product
1	$\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$	$\text{Fe}(\text{DS})_3$	25	1	80	Yellow
2	$\text{Sr}(\text{NO}_3)_2$	$\text{Sr}(\text{DS})_2$	25	0.5	85	White
3	$\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$	$\text{Al}(\text{DS})_3$	25	0.5	80	White
4	AgNO_3	$\text{Ag}(\text{DS})$	100	5	52	Black
5	$\text{Pb}(\text{NO}_3)_2$	$\text{Pb}(\text{DS})_2$	25	1	90	White
6	$\text{Cd}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$	$\text{Cd}(\text{DS})_2$	100	12	0	–
7	$\text{Mg}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$	$\text{Mg}(\text{DS})_2$	100	12	0	–
8	$\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$	$\text{Cu}(\text{DS})_2$	100	12	0	–
9	$\text{Zr}(\text{NO}_3)_4$	$\text{Zr}(\text{DS})_4$	25	0.5	87	White
10	$(\text{NH}_4)_2\text{Ce}(\text{NO}_3)_6$	$\text{Ce}(\text{DS})_4$	25	5	65	Yellow
11	$\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$	$\text{Ca}(\text{DS})_2$	25	1	88	White
12	$\text{Zn}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$	$\text{Zn}(\text{DS})_2$	100	12	0	–
13	$\text{Mn}(\text{NO}_3)_2 \cdot 2\text{H}_2\text{O}$	$\text{Mn}(\text{DS})_2$	100	12	0	–
14	$\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$	$\text{Co}(\text{DS})_2$	100	12	20	Grey
15	$\text{Cr}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$	$\text{Cr}(\text{DS})_3$	25	1	72	Green

Table 2
Synthesis of **3b** by different metal dodecyl sulfates



Entry	Salt	Time (h)	Yield (%) ^a
1	$\text{Fe}(\text{DS})_3$	4.75	85
2	$\text{Sr}(\text{DS})_2$	12	40
3	$\text{Al}(\text{DS})_3$	5.5	87
4	$\text{Ag}(\text{DS})_3$	12	50
5	$\text{Pb}(\text{DS})_2$	12	80
6	NaDS	12	50
7	$\text{Cr}(\text{DS})_3$	4.5	81
8	$\text{Zr}(\text{DS})_4$	4 (12) ^b	83 (80) ^b
9	$\text{Ce}(\text{DS})_4$	7.5	91
10	$\text{Ca}(\text{DS})_2$	12	40

^a Yields refer to isolated pure products.

^b Recovered catalyst.

Table 3
Zr(DS)₄ Catalyzed synthesis of quinolines in water at reflux conditions^a

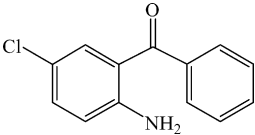
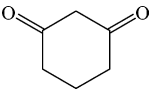
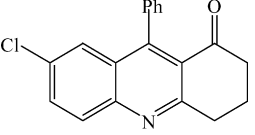
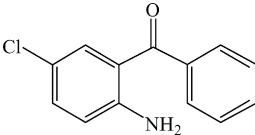
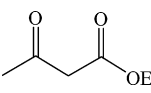
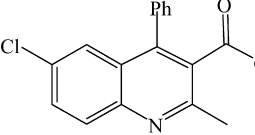
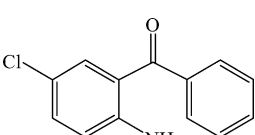
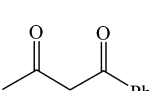
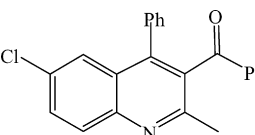
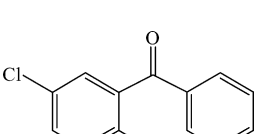
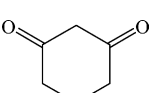
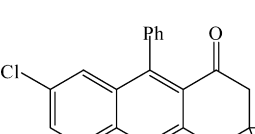
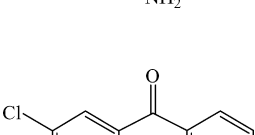
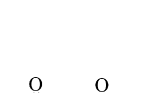
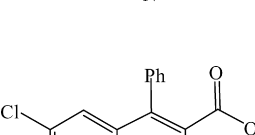
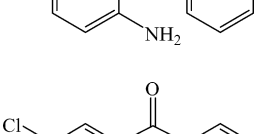

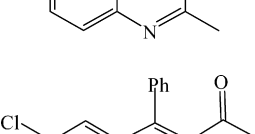
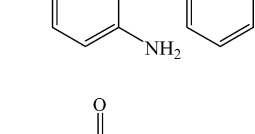

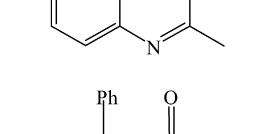
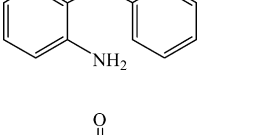
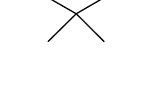
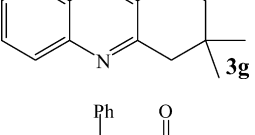
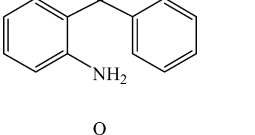
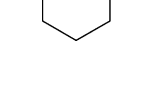
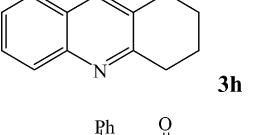
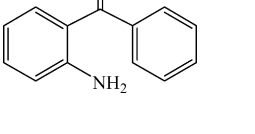
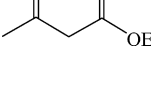
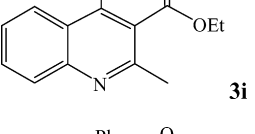
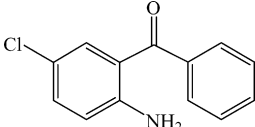
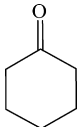
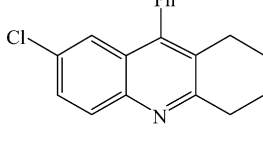
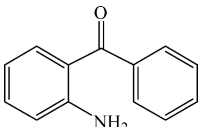
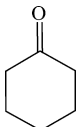
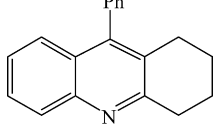
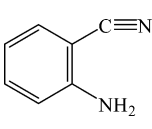
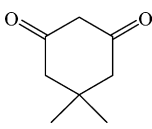
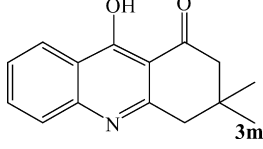
Entry	Substrate 1	Ketone 2	Quinoline 3	Time (h)	Yield (%) ^b
1			 3a	0.5 (4) ^c	80 (80) ^c
2			 3b	3.15 (3.15) ^c	85 (85) ^c
3			 3c	0.75 (3.5) ^c	63 (62) ^c
4			 3d	0.5 (4.25) ^c , (4.5) ^d , (7) ^e , (6) ^f	88 (88) ^c , (70) ^d , (83) ^e , (79) ^f
5			 3e	4.45	74
6			 3f	1.45	68
7			 3g	0.5	93
8			 3h	0.8	98
9			 3i	3	72
10			 3j	3.45	80

Table 3 (Continued)

Entry	Substrate 1	Ketone 2	Quinoline 3	Time (h)	Yield (%) ^b
11 ^g			 3k	6	79
12 ^g			 3l	6	90
13			 3m	24	50

^a The reactions were performed in H₂O and in the presence of Zr(DS)₄ (10% eq.) unless otherwise stated.

^b Yields refer to isolated pure products.

^c H₂O:EtOH (2:1) as a solvent.

^d CHCl₃ as a solvent.

^e 96% EtOH as a solvent.

^f *n*-Hexane as a solvent.

^g Five equivalents of cyclohexanone was used.

activate the substrate molecules and as a surfactant to increase the concentration of organic reactants to form colloidal particles.

In conclusion, we report the preparation of new types of combined Lewis acid-surfactant catalysts (LASC) and their applications in the synthesis of quinoline derivatives by Friedlander protocol in aqueous media. Among the described metal dodecyl sulfates, Zr(DS)₄ was superior to others and could be recovered and reused in aqueous media.

1. Experimental

1.1. General

All chemicals were purchased from Merck and Fluka companies and used without any further purification. ¹H NMR and ¹³C NMR spectra were recorded at 90 and 22.5 MHz, respectively, using a convenient deuterated solvent (reported below) and the residual peak as internal standard, or TMS in the case of CDCl₃. Melting points are not corrected. Thin layer chromatography was performed on precoated silica gel aluminum plates. Quinolines were characterized by comparison of their spectral (IR, ¹H NMR and ¹³C NMR) and physical data with those of authentic samples [5,6].

1.1.1. Preparation of Zr(DS)₄ from Zr(NO₃)₄ and sodium dodecyl sulfate

To a stirring solution of sodium dodecyl sulfate (40 mmol, 11.51 g) in distilled water (300 ml) was added a solution of Zr(NO₃)₄ (10 mmol, 2.83 g) in water (100 ml) at room temperature. A white precipitate appeared immediately and the mixture was stirred for another 30 min. The white solid was separated

by filtration and washed with water (2 × 50 ml). The isolated solid residue was dried under reduced pressure and Zr(DS)₄ as a white powder was obtained in 87% yield (10 g). mp 168 °C (dec); FT-IR (KBr): ν 1378, 1177, cm⁻¹. ¹H NMR (90 MHz, DMSO-d₆): δ : 3.344 (t, 8H), 0.95–1.52 (m, 80H), 0.816 (t, 12H), ¹³C NMR (22.5 MHz, DMSO-d₆): δ 66.175, 61.155, 32.814, 31.642, 29.396, 29.056, 25.825, 22.383, 14.02.

1.1.2. Typical experimental procedure

A mixture of 2-amino-5-chlorobenzophenone (233 mg, 1 mmol), and ethyl acetoacetate (195 mg, 1.5 mmol), was added to a suspension of Zr(DS)₄ (0.115 g, 0.1 mmol) in water (5 ml) under reflux conditions. Reaction was completed after 3.15 h. EtOAc (3 × 10 ml) was added to the reaction mixture and organic phase was separated and dried over anhydrous Na₂SO₄. After removing of solvent, 6-chloro-3-(ethylformate)-2-methyl-4-phenylquinoline **3b** as white crystals was obtained in 85% yield (275 mg). Crude product was recrystallized in ethanol. For recovering the catalyst, aqueous phase and organic phase were centrifuged together which a triphase system (organic, aqueous and solid phase) was obtained. The solid phase was separated and dried under reduced pressure and reused.

1.2. Spectral data for quinolines

(3a): Yellow solid; mp 185 °C; FT-IR (KBr): ν 3024, 2956, 1684, 1551, 1476, 1075, 838, 697 cm⁻¹. ¹H NMR (90 MHz, CDCl₃): δ : 2.24 (m, 2H), 2.71 (t, 2H), 3.36 (t, 2H), 7.20–7.95 (m, 8H). MS: *m/z* 307.

(3b): Yellow solid; mp 101 °C; FT-IR (KBr): ν 3076, 2977, 2930, 1719, 1602, 1564, 1479, 1381, 1308, 1216, 1162, 1069,

885, 797, 609 cm^{-1} . $^1\text{H NMR}$ (90 MHz, CDCl_3): δ : 0.95 (t, 3H), 2.78 (s, 3H), 4.03 (q, 2H), 7.46–7.96 (m, 8H). MS: m/z 325.

(3c): White solid; mp 217 °C; FT-IR (KBr): ν 3390, 1672, 1569, 1480, 1226, 1022, 967, 831, 785, 699 cm^{-1} . $^1\text{H NMR}$ (90 MHz, CDCl_3): δ : 2.62 (s, 3H), 7.25–8.03 (m, 13H). $^{13}\text{C NMR}$ (22.5 MHz, CDCl_3): δ : 23.95, 124.97, 126.12, 128.22, 128.51, 129.19, 129.93, 130.65, 130.91, 132.45, 133.62, 134.20, 136.99, 144.71, 146.28, 155.07, 197.24. MS: m/z 357.

(3d): Yellow solid; mp 211 °C; FT-IR (KBr): ν : 3074, 2952, 2866, 1696, 1554, 1477, 1384, 1297, 1198, 1079, 837, 699 cm^{-1} . $^1\text{H NMR}$ (90 MHz, CDCl_3): δ : 1.15 (s, 6H), 2.56 (s, 2H), 3.25 (s, 2H), 7.21–7.95 (m, 8H). MS: m/z 323.

(3e): Yellow solid; mp 135 °C; FT-IR (KBr): ν : 3455, 3049, 2948, 1736, 1559, 1481, 1378, 1283, 1214, 1161, 1067, 828, 759, 712 679 cm^{-1} . $^1\text{H NMR}$ (90 MHz, CDCl_3): δ : 2.76 (s, 3H), 3.58 (s, 3H), 7.47–7.96 (m, 9H). MS: m/z 313.

(3f): Yellow solid; mp 157 °C; FT-IR (KBr): ν : 3076, 2977, 1720, 1561, 1479, 1380, 1219, 1068, 840, 711 cm^{-1} . $^1\text{H NMR}$ (90 MHz, CDCl_3): δ : 2.00 (s, 3H), 2.68 (s, 3H), 7.35–7.95 (m, 8H). MS: m/z 297.

(3g): Yellow solid; mp 192 °C; FT-IR (KBr): ν : 3067, 2936, 2868, 1684, 1609, 1558, 1483, 1389, 1370, 1345, 1285, 1218, 1125, 1051, 842, 770, 695 cm^{-1} . $^1\text{H NMR}$ (90 MHz, CDCl_3): δ : 1.15 (s, 6H), 2.55 (s, 2H), 3.27 (s, 2H), 7.18–8.02 (m, 9H). $^{13}\text{C NMR}$ (22.5 MHz, CDCl_3): δ : 28.18, 31.99, 48.25, 54.04, 126.24, 127.26, 127.91, 127.98, 128.44, 131.27, 137.46, 148.91, 150.71, 160.94, 197.48. MS: m/z 300.

(3h): Yellow solid; mp 159 °C; FT-IR (KBr): ν : 3054, 2948, 2874, 1689, 1552, 1484, 1392, 1218, 1157, 1022, 922, 770, 757, 705, 612 cm^{-1} . $^1\text{H NMR}$ (90 MHz, CDCl_3): δ : 2.21 (q, 2H), 2.69 (t, 2H), 3.36 (t, 2H), 7.21–8.02 (m, 9H). $^{13}\text{C NMR}$ (22.5 MHz, CDCl_3): δ : 20.86, 34.09, 40.09, 123.38, 125.90, 127.00, 127.67, 128.09, 131.11, 137.20, 148.21, 150.65, 161.73, 197.08.

(3i): Yellow solid; mp 99 °C; FT-IR (KBr): ν : 3064, 2978, 2928, 1712, 1561, 1402, 1297, 1237, 1182, 1070, 872, 767 cm^{-1} . $^1\text{H NMR}$ (90 MHz, CDCl_3): δ : 0.86 (t, 3H), 2.71 (s, 3H), 3.93 (q, 2H), 7.35–7.95 (m, 9H). MS: m/z 291.

(3j): White solid; mp 107 °C; FT-IR (KBr): ν : 3064, 2950, 1731, 1582, 1486, 1426, 1392, 1295, 1229, 1170, 1067, 1029, 967, 761, 700, 601 cm^{-1} . $^1\text{H NMR}$ (90 MHz, CDCl_3): δ : 2.78 (s, 3H), 3.55 (s, 3H), 7.41–8.03 (m, 9H). MS: m/z 277.

(3k): Yellow solid; mp 165 °C; FT-IR (KBr): ν : 3060, 2945, 2858, 1600, 1569, 1477, 1440, 1349, 1266, 1165, 1075, 937, 831, 818, 756, 708, 617 cm^{-1} . $^1\text{H NMR}$ (90 MHz, CDCl_3): δ : 1.89 (m, 4H), 2.58 (t, 2H), 3.17 (t, 2H), 7.27–7.89 (m, 8H).

(3l): Yellow solid; $^1\text{H NMR}$ (90 MHz, CDCl_3): δ : 1.86 (m, 4H), 2.59 (t, 2H), 3.20 (t, 2H), 7.26–7.98 (m, 9H). $^{13}\text{C NMR}$ (22.5 MHz, CDCl_3): δ : 22.64, 22.76, 27.74, 33.99, 125.09, 125.47, 126.40, 127.44, 128.01, 128.32, 128.85, 136.88, 146.13, 158.71.

(3m): Yellow solid; mp 176 °C; FT-IR (KBr): ν : 3216, 3016, 2946, 1579, 1519, 1370, 1275, 1247, 1156, 1099, 929, 857, 819, 775, 758, 657 cm^{-1} . $^1\text{H NMR}$ (90 MHz, CDCl_3): 1.05 (s, 6H), 2.17 (s, 2H), 2.35 (s, 2H), 5.40 (s, 1H), 6.90–7.42 (m, 4H); $^1\text{H NMR}$ (90 MHz, CDCl_3 plus few drops of D_2O): 1.13 (s, 6H), 2.25 (s, 2H), 2.43 (s, 2H), 4.70 (due to produced water), 7.26–7.51 (m, 4H). $^{13}\text{C NMR}$ (22.5 MHz, CDCl_3): δ : 28.22,

29.58, 32.89, 43.21, 50.38, 100.68, 125.68, 133.81, 141.44, 159.73, 198.34. MS: m/z 357.

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