

Room temperature aqueous Paal–Knorr pyrrole synthesis catalyzed by aluminum tris(dodecyl sulfate)trihydrate

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Abstract This article reports a novel procedure to prepare pyrroles using a modification of the Paal–Knorr reaction. Water is a safe solvent meeting environmental considerations, but most organic substrates are not soluble in water. A possible solution to improve the solubility of substrates is the use of surface-active reagents that can form micelles. For instance, combined Lewis acid–surfactant catalyst acts both as a Lewis acid to activate the substrate molecules and as a surfactant to form emulsions in water. Here, we prepared and used aluminum tris(dodecyl sulfate)trihydrate to condense various amines to 2,5-hexadione at room temperature. The sole solid pyrrole was separated by a simple filtration. Our findings thus show a novel and improved modification of the Paal–Knorr reaction in terms of mild reaction conditions and clean reaction profiles, using a simple workup procedure and improved yields with excellent chemo-selectivity.

Keywords Aluminum tris (dodecyl sulfate) trihydrate · Pyrrole · Paal–Knorr · Click reaction · Water

Introduction

Water is a desirable solvent for reasons of cost, safety, and environmental concerns. Moreover, environmental consciousness imposes the use of water for organic processes from both industrial and academic points of views (Kolb et al. 2001; Li and Chan 2007; Narayan et al. 2005). Water possesses unique physical and chemical properties, and its

use could enhance the reactivity and selectivity generally achieved in classical organic solvents (Rideout and Breslow 1980; Otto and Engberts 2003). However, organic solvents are still used instead of water for mainly two reasons; first, most organic substrates are not soluble in water, and, as a result, water cannot function as a reaction medium. Second, many reactive substrates, reagents, and catalysts are sensitive to water and are decomposed or deactivated in aqueous media. A possible way to improve the solubility of substrates is the use of surface-active reagents that can form micelles (Cramer and Truhlar 1994) or vesicular structures. The use of micellar and vesicle-forming surfactants as catalysts has been investigated in detail for different reactions in aqueous solutions (Tas Cioglu et al. 1996; Dwars et al. 1998; Goedheijt et al. 2000). Recently, ‘Lewis acid–surfactant combined catalyst’ shows high efficiency in various organic transformations as a new type of catalyst. These reactions are promoted in water without organic cosolvents (Manabe et al. 2001; Shiri and Zolfigol 2009; Firouzabadi et al. 2005, 2007). This kind of catalyst acts both as a Lewis acid to activate the substrate molecules and as a surfactant to form emulsions in water.

The pyrrole ring system is a useful structural element in medicinal chemistry (Jones and Bean 1977) and has found broad application in drug development for the treatment of antibacterial, antiviral, anti-inflammatory, antitumoral, and antioxidant materials (Furstner 2003). They are a highly versatile class of intermediates in the synthesis of natural products as well as in heterocyclic chemistry (Boger et al. 1999) and very useful in materials science (Domingo et al. 2001). It is therefore not surprising that many synthetic methods have been developed for the preparation of these compounds (Ferreira et al. 2001). Despite these huge developments, the Paal–Knorr reaction is considered to be

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the most attractive method for the synthesis of pyrroles; wherein 1,4-dicarbonyl compounds are converted to pyrroles by the a reaction with primary amines in the presence of various promoting agents (Jones and Bean 1977; Darabi et al. 2012a; Curini et al. 2003; Banik et al. 2004; Wang et al. 2004; Ballini et al. 2000; Minetto et al. 2005; Abid et al. 2006; Chen et al. 2006; Banik et al. 2005; Yuan et al. 2010; Chen et al. 2008; Luo et al. 2008). However, some of these methods often suffer from certain drawbacks such as hazardous organic solvents, high cost, use of stoichiometric or relatively expensive catalysts, tedious workup leading to the generation of large amounts of toxic metal-containing wastes, and low yields of products. Moreover, the high cost and susceptibility to moisture would be a major concern in the industrial applications of a metal salt. Hence, the development of facile and environment friendly methods (using no organic solvent in reaction and/or in workup procedure) for this purpose would be of value and is still required for green organic synthesis.

In the course of our investigations to develop new synthetic reactions in water, recently, we have successfully applied the micellar solution of sodium dodecyl sulfate for hetero-Michael, and Mannich reaction under neutral conditions and prepared sulfoxides via green oxidation of sulfides in aqueous hydrogen peroxide (Jafari et al. 2009a, b; Tamaddon et al. 2010). Herein, we report the use of

aluminum dodecyl sulfate trihydrate as a stable and effective Lewis acid-surfactant combined catalyst (2.5 mol%) for the chemoselective condensation of amines with hexane-2,5-dione in water.

Experimental

General remarks

The chemical materials were purchased from Merck and Fluka Chemical Companies. All the products were known and characterized by comparing their physical data with those reported in the literature. Infrared spectra were run on a Shimadzu model 8,300 Fourier Transform Infrared spectrophotometer. Nuclear magnetic resonance spectra were recorded on a Bruker Avance DPX-400 and 500. The purity of the products and the progress of the reactions were determined and established by thin layer chromatography on silica-gel polygram SILG/UV₂₅₄ plates or by a Shimadzu Gas Chromatograph-14A instrument with a flame ionization detector.

General procedure for the condensation of amine to 2,5-hexanedione

Al(dodecyl sulfate)₃·3H₂O (0.041 g, 0.025 mmol) was added to a mixture of amine (2.0 mmol) and 2,5-hexanedione (0.25 g, 2.2 mmol) in 2 mL water. The reaction mixture was stirred at room temperature for an appropriate time (Table 1, 2). During the stirring, a desired solid product separated out. After the completion of the reaction (monitored by thin layer chromatography), water (5 mL) was added to the reaction mixture. The solid product was collected by a simple filtration, washed with water (10 mL), and air-dried to provide the sole product as a white or cream powder.

The selected spectral and analytical data for new compounds: 1-(4-fluorobenzyl)-2,5-dimethyl-1H-pyrrole (Table 2, entry 13): white solid, mp 54–55 °C. FT-IR: ν_{\max} (neat) = 3,010, 2,934, 1,605, 1,508, 1,443, 1,409, 1,347, 844 cm⁻¹, ¹HNMR (500 MHz, CDCl₃): δ = 2.18 (s, 6H), 5.02 (s, 2H), 5.91 (s, 2H), 6.89 (dd, J = 8.6, 5.4 Hz, 2H), 7.03 (dd, J = 8.6, 8.6 Hz, 2H).ppm, ¹³CNMR (125 MHz, CDCl₃): δ = 12.8, 46.5, 106.0, 115.9, 116.1, 127.6, 127.7, 128.3, 134.6, 134.7, 161.4, 163.3 ppm. N-(2-(2,5-dimethyl-1H-pyrrol-1-yl) ethyl) benzeneamine (Table 2, entry 14): white solid, mp 70–71 °C, FT-IR: ν_{\max} (neat) = 3,354, 2,913, 1,600, 1,509, 1,444, 1,350, 1,325, 773, 750 cm⁻¹, ¹HNMR (500 MHz, CDCl₃): δ = 2.27 (s, 6H), 3.45 (t, J = 6.6 Hz, 2H), 4.00 (s, NH), 4.02 (t, J = 6.6 Hz, 2H), 5.85 (s, 2H), 6.65 (d, J = 7.7 Hz, 2H), 6.78 (t, J = 7.4 Hz, 1H), 7.24 (dd, J = 8.3, 7.4 Hz, 2H) ppm, ¹³CNMR (125 MHz, CDCl₃): δ = 13.0,

Table 1 Optimization of reaction conditions

Entry	Catalyst (mol%)	Time (h)	Temperature (°C)	Yield (%)
1	None	24	r.t.	10
2	Triton × 100 (2.5)	24	r.t.	40
3	CTAB ^a (2.5)	24	r.t.	30
4	SDS ^b (CMC ^c)	24	r.t.	45
5	SDS (CMC)	24	50	53
6	SDS (CMC)	14	60	55
7	SDS (CMC)	12	80	70
8	SDS (5)	24	r.t.	60
9	SDS (2.5)	24	r.t.	50
10	LiDS ^d (2.5)	24	r.t.	55
11	Al(DS) ₃ ^e (2.5)	9	r.t.	93
12	Al(DS) ₃ (5)	8	r.t.	92
13	Al(DS) ₃ (1)	12	r.t.	70
14	Mg(DS) ₂ ^f (2.5)	24	r.t.	60

^a Cetyl trimethylammonium bromide (CTAB)

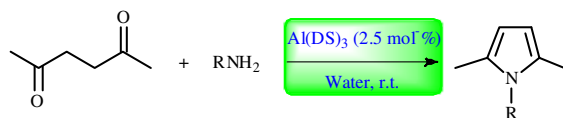
^b Sodium dodecyl sulfate (SDS)

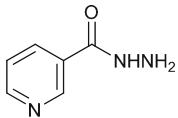
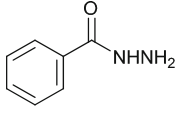
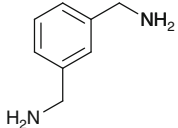
^c Critical micelle concentration (CMC)

^d Lithium dodecyl sulfate (LiDS)

^e Aluminum tris (dodecyl sulfate) trihydrate (Al(DS)₃·3H₂O)

^f Magnesium bis (dodecyl sulfate) (Mg(DS)₂)

Table 2 Al(dodecyl sulfate)₃·3H₂O catalyzed synthesis of N-substituted pyrroles in water at room temperature^a


Entry	RNH ₂	Time (h)	Yield (%)	m.p. (°C) [reported]	References
1	PhNH ₂	9	93	47–49 (50)	Luo et al. (2008)
2	3-Me-C ₆ H ₄ NH ₂	12.5	92	50–51	Yuan et al. (2010)
3	4-Me-C ₆ H ₄ NH ₂	12	93	44–45	Yuan et al. (2010)
4	4-MeO-C ₆ H ₄ NH ₂	8	98	55–56 (59–60)	Curini et al. (2003)
5	3,4-diMeO-C ₆ H ₄ NH ₂	13	90	51–52	
6	2-OH-C ₆ H ₄ NH ₂	36	94	93–94	Chen et al. (2006)
7	4-Cl-C ₆ H ₄	9.25	96	47–48	Yuan et al. (2010)
8	3-Cl-C ₆ H ₄ NH ₂	12.5	98	48–49	Chen et al. (2008)
9	4-Br-C ₆ H ₄ NH ₂	16	95	72–73	Yuan et al. (2010)
10	2-Ph-C ₆ H ₄ NH ₂	40	95	97–98	Abid et al. (2006)
11	1-Naphthylamine	11	98	118–119 (118)	Banik et al. (2004)
12	PhCH ₂ NH ₂	3	96	42–43 (42–44)	Curini et al. (2003)
13	4-F-C ₆ H ₄ CH ₂ NH ₂	3.5	97	54–55	Chen et al. (2006)
14	PhNHCH ₂ CH ₂ NH ₂	4	98	70–71	
15	Pyridin-2-ylmethylNH ₂	5	90	53–54	
16		15	90	137–138	
17		30	96	180–181	
18		3	95	89–91	

^a Reaction conditions: water (2 mL), hexane-2,5-dione (2.2 mmol), aniline (2 mmol)

43.2, 44.3, 106.1, 113.1, 118.3, 128.2, 129.9, 147.8 ppm. 2-((2,5-dimethyl-1H-pyrrol-1-yl)methyl)pyridine (Table 2, entry 15): white solid, mp 53–54 °C, FT-IR: ν_{\max} (neat) = 3,040, 2,932, 1,633, 1,591, 1,571, 1,440, 1,350, 768, 751 cm^{-1} , ¹HNMR (500 MHz, CDCl₃): δ = 2.17 (s, 6H), 5.16 (s, 2H), 5.91 (s, 2H), 6.47 (d, J = 7.8 Hz, 1H), 7.19 (dd, J = 7.1, 5.1 Hz, 1H), 7.61 (ddd, J = 7.6, 7.6, 1.7 Hz, 1H), 8.60 (d, J = 4.6 Hz, 1H) ppm, ¹³CNMR (125 MHz, CDCl₃): δ = 12.8, 49.2, 106.2, 120.3, 122.5, 128.3, 137.7, 149.7, 159.0 ppm. N-(2,5-dimethyl-1H-pyrrol-1-yl) nicotinamide (Table 2, entry 16): white solid, mp 137–138 °C, FT-IR: ν_{\max} (neat) = 3,218, 2,922, 1,671, 1,537, 1,518, 1,491, 1,322, 1,288, 769, 748 cm^{-1} , ¹HNMR (500 MHz, CDCl₃): δ = 2.15 (s, 6H), 5.85 (s, 2H), 7.73 (d, J = 4.2 Hz, 2H), 8.77 (s, 2H), 9.20 (s, 1H) ppm, ¹³CNMR (125 MHz, CDCl₃): δ = 11.6, 105.0, 121.8, 128.2, 150.8, 165.2 ppm. 1-(3-((2,5-dimethyl-1H-pyrrol-1-yl)

methyl) benzyl)-2,5-dimethyl-1H-pyrrole (Table 2, entry 18) white solid, mp 89–91 °C, FT-IR: ν_{\max} (neat) = 3,000, 2,930, 1,607, 1,520, 1,443, 1,407, 1,325, 1,301, 974, 751 cm^{-1} , ¹HNMR (500 MHz, CDCl₃): δ = 2.16 (s, 12H), 4.99 (s, 4H), 5.89 (s, 4H), 6.58 (s, 1H), 6.74 (d, J = 7.5 Hz, 2H), 7.24 (t, J = 7.5 Hz, 1H) ppm, ¹³CNMR (125 MHz, CDCl₃): δ = 12.8, 47.1, 105.9, 123.5, 124.8, 128.3, 129.7, 139.5 ppm.

Result and discussion

To find the optimal conditions, the reaction of aniline (2 mmol), hexane-2,5-dione (2.2 mmol) as a model reaction under various reaction conditions was studied in 2 mL water (Table 1). At first, the model reaction was performed in pure water. The reaction proceeded sluggishly and after

Scheme 1

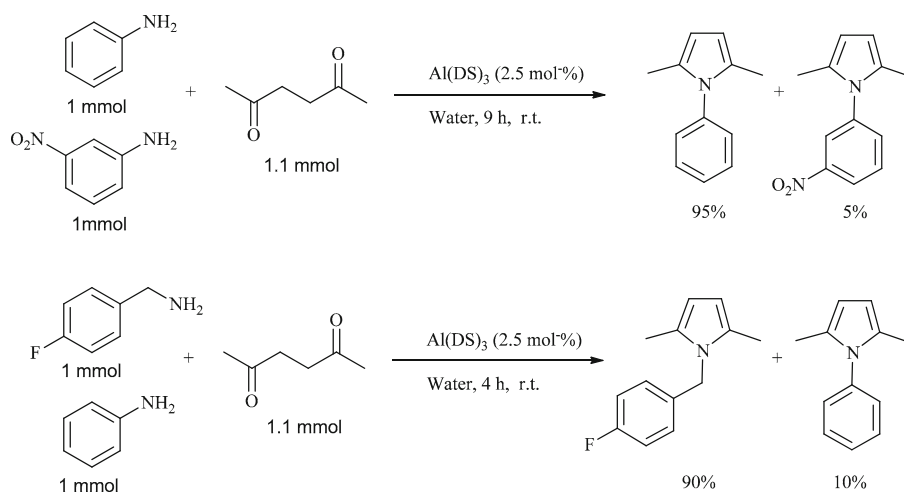


Table 3 Comparison of the catalytic activity of Al(dodecyl sulfate)₃·3H₂O with those of different catalysts for the reaction of aniline with hexane-2,5-dione

Entry	Solvent	Catalyst (mol %)	Loading (mol %)	Time (h)	Yield (%)	Ref.
1	CH ₂ Cl ₂	Bi(NO ₃) ₃	5	10	96	Banik et al. (2005)
2	CH ₂ Cl ₂	Montmorillonite, KSF	1g	10	95	Banik et al. (2004)
3	Hexane	SbCl ₃ /SiO ₂	0.1	1	91	Darabi et al. (2012b)
4	EtOH	Vitamin B ₁	5	1	90	Darabi et al. (2012a)
5	H ₂ O	Al(DS) ₃ ·3H ₂ O	2.5	9	93	This work

some prolonged reaction time (24 h), N-phenyl pyrrole was produced in only a 10 % yield. The problem with the use of water in the reaction (this reaction in water as we have observed) was the formation of a gummy mass in the reaction media. Then, to solve this problem, a similar reaction was performed in the presence of various surfactants and surfactant-type Lewis acid catalyst (Table 1, entries 2–14). We observed a drastic rate of enhancement when performing this reaction in water in the presence of 2.5 mol % of aluminum tris(dodecyl sulfate)trihydrate (Al(dodecyl sulfate)₃·3H₂O) to produce N-phenyl pyrrole in 93 % yield after 9 h at room temperature. Similar reactions in the presence of other surfactants (Table 1, entries 2–10, 14); sodium dodecyl sulfate, lithium dodecyl sulfate, magnesium bis(dodecyl sulfate), cetyl trimethylammonium bromide, and triton X-100 did not proceed to completion even after 24 h, and the desired adduct was produced in 50, 55, 60, 30, and 40 % yields, respectively together with unreacted starting materials. These results showed that a combination of a Lewis acid moiety (cationic part) and a surfactant (anionic part) in Al(dodecyl sulfate)₃·3H₂O is important for reactions to proceed in water. The Lewis acid part of the catalyst activates the substrate molecules and the surfactant part, affecting the solubility of the substrates in water at the same time. When these two factors are in play together, a drastic enhancement is observed of the rates and the yields of the products.

After this success, in order to show the general applicability of the method, the reaction of structurally diverse amines with hexane-2,5-dione was studied under similar reaction conditions. By this method, aromatic and aliphatic amines easily reacted with hexane-2,5-dione to produce N-substituted pyrroles in high to excellent yields. Aliphatic amines reacted more efficiently than aromatic ones to give N-alkyl pyrroles in excellent yields. In the case of anilines having an electron-donating group, the corresponding products were obtained in quantitative yields. It is to mention that sterically hindered 2-phenylaniline gave the corresponding product in an excellent yield (Table 2, entry 10). Furthermore, new N-benzamido pyrroles were synthesized easily in high to excellent yields (Table 2, entries 16, 17). Aliphatic amines, such as N-phenylethane-1,2-diamine, pyridin-2-ylmethanamine, and benzylamine, gave the corresponding pyrroles in high yield under the same conditions. In all the cases, the pyrrole products were precipitated as the reaction proceeded, and the pure product was isolated by a simple filtration without using any chromatography or cumbersome reaction workup. This extra simple procedure opened the route for an entirely green highly efficient pyrrole synthesis (a click Paal-Knorr pyrrole synthesis) in water.

Through this study, we also investigated the applicability of the method for the preparation of bispyrrole via the reaction of α,α' -diamino-*m*-xylene (1 mmol) and hexane-2,5-dione (2.2 mmol) under same conditions. The reactions

were progressed well, and the desired bis-pyrrole was produced in an excellent yield (Table 2, Entry 18). The desired bis-pyrroles were precipitated as the reaction proceeded. The sole products were achieved in a 95 % yield after addition of water to the reaction mixture and isolated by a simple filtration. Bispyrroles are interesting compounds, for example, they can be employed as the monomers for the preparation of macrocyclic or polymeric compounds with a pyrrole structure.

Selectivity of the method is important for the total synthesis of complex organic molecules. In order to show the high selectivity of the method, we performed two competitive reactions for the Paal–Knorr pyrrole synthesis using Al(dodecyl sulfate)₃ as a catalyst in water at room temperature. Using this green catalytic system, the highly selective conversion of aliphatic amine in the presence of aromatic ones was observed. We also studied the competitive reactions of aniline versus 3-nitroaniline. This reaction also proceeded with high selectivity to show the importance of electronic effects upon the Al(dodecyl sulfate)₃ catalyzed reaction. The results of this excellent chemo-selectivity are shown in Scheme 1.

In order to show the merits of the presented protocol for the Paal–Knorr pyrrole synthesis, we have compared the results obtained using Al(dodecyl sulfate)₃·3H₂O with some of those recently reported in the literature for the reaction of aniline with hexane-2,5-dione as tabulated in Table 3.

Conclusion

In conclusion, this procedure offers several advantages for the Paal–Knorr pyrrole synthesis. Among them, one can mention the use of water as a green solvent, low loading of cheap Al(dodecyl sulfate)₃·3H₂O as a catalyst, high yields, excellent chemo-selectivity, and cleanliness. In addition, the self separation of the products (Click Chemistry) which are insoluble in water is noteworthy; they can be easily isolated by a simple filtration. This simple work-up procedure is of practical importance, especially for large-scale operations.

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