One-Pot Synthesis of N-Substituted Pyrroles Catalyzed by Polystyrene-Supported Aluminum Chloride as a Reusable Heterogeneous Lewis Acid Catalyst

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ABSTRACT: A convenient and efficient procedure is presented for the one-pot synthesis of N-substituted pyrroles by condensation of 2,5-hexandione and amines or diamines in the presence of cross-linked polystyrene-supported aluminum chloride (PS/AlCl₃) as a highly active and reusable heterogeneous Lewis acid catalyst. This polymeric solid acid catalyst is stable and can be easily recovered and reused without appreciable change in its efficiency. © 2010 Wiley Periodicals, Inc. Heteroatom Chem 22:85–90, 2011; View this article online at wileyonlinelibrary.com. DOI 10.1002/hc.20661

INTRODUCTION

Pyrroles are important heterocyclic compounds displaying remarkable pharmacological properties such as antibacterial, antiviral, anti-inflammatory, antitumoral, and antioxidant activities [1]. The pyrrole moiety is found in many naturally occurring compounds such as heme, chlorophyll, and vitamin B_{12} [2]. Pyrroles are also present in various bioactive drug molecules such as atorvastatin, anti-inflammatory and antitumor agents, and immuno-

suppressants [3,4]. In view of their high significance, many methodologies have been developed for the construction of the pyrrole skeleton [5]. Among them, the Paal-Knorr synthesis remains the most useful preparative method for generating pyrroles [6]. In recent years, a variety of reagents such as K10 $clay [7], Bi(NO_3)_3 \cdot 5H_2O [8], Dy(OTf)_3 [9], PMA/SiO_2$ [10], β-CD [11], aluminum oxide [12], acidic resin (D001) [13], zinc tetrafluoroborate [14], and I_2 [15] under reflux conditions and at room temperature have been utilized for the synthesis of N-substituted pyrroles. In addition, the above-mentioned cyclocondensation process could proceed in ionic liquid [16] or ultrasonic and microwave irradiation [17]. Many of these procedures involve the use of expensive reagents, metal triflates, and extended reaction times and produce a huge amount of corrosive, toxic waste products and tedious workup and the use of extra energy source, such as high-power microwave irradiation or ultrasound. So, a milder, nonhazardous, recyclable, and ecofriendly organic catalyst is still in demand.

One of the main themes of contemporary synthetic organic chemistry is the development of atomeconomic and environmentally benign catalytic systems [18]. In this context, the development of heterogeneous catalysis is of prime importance not only from the economic point of view but also due to the easy workup procedures involved in the separation of products from a catalyst. In recent years, a large number of polymer-supported Lewis

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SCHEME 1 PS/AICl₃-catalyzed synthesis of pyrroles.

acids have been prepared by immobilization of the catalysts on polymers via coordination or covalent bonds and are used in many organic transformations [19–22]. Such polymeric catalysts are usually as active as their homogeneous counterparts while having the advantages of recyclability, greater selectivity, enhanced stability, easier handling, nontoxicity, and noncorrosiveness. The most frequently used polymeric support is polystyrene; its hydrophobic nature protects water-sensitive Lewis acids from hydrolysis by atmospheric moisture until it is suspended in an appropriate solvent where it can be used in a chemical reaction [21,23]. Recently, polystyrenesupported aluminum chloride has emerged as a promising heterogeneous Lewis acid catalyst for acid-catalyzed reactions, such as Friedel-Crafts acylation and sulfonylation reactions, tetrahydropyranylation of alcohols and phenols, and the synthesis of bis-indolylmethanes [19a,19b,24].

In continuation of our interest in the synthesis of organic compounds from condensation of phenols with dialdehydes [25] and our ongoing research on the use of heterogeneous Lewis acid catalysts, herein, we wish to report an efficient and environmentally benign method for the synthesis of substituted pyrrole derivatives catalyzed by polystyrene-supported aluminum chloride (PS/AlCl₃) (Scheme 1). To the best of our knowledge, polystyrene-supported aluminum chloride has not been used in the synthesis of pyrroles.

RESULTS AND DISCUSSION

For our investigations, cross-linked PS/AlCl₃ was prepared by addition of anhydrous aluminum chloride to polystyrene (8% divinylbenzene) in carbon disulfide under reflux conditions [21b]. The capacity of the polymeric catalyst was 0.48 mmol AlCl₃ per gram catalyst [19]. Although AlCl₃ is a watersensitive, corrosive, and environmentally harmful compound, PS/AlCl₃ is a stable and water-tolerant species. This polymeric catalyst is easy to prepare, stable in air for a long time without any change, easily recycled and reused without appreciable loss of its activity.

In our initial research, to determine the most appropriate reaction conditions and evaluate the catalytic efficiency of the PS/AlCl₃ catalyst, the condensation reaction of aniline (1 mmol) with 2,5hexandione (1 mmol) was carried out in different solvents. Among the various solvents screened, CH₃CN was the most effective reaction media (Table 1, entry 6). In addition, we also studied influence of the amount of PS/AlCl₃ on the reaction yields. We found that the use of 15 mol% of PS/AlCl₃ was sufficient to progress the reaction and gave **3a** in 90% yield in 75 min under reflux conditions and also an increase in the amount of catalyst did not improve the yield (Table 1, entry 7). A comparative reaction was examined with aniline and 2,5-hexandione in the absence of catalyst, and no product was observed (Table 1, entry 11). The immobilized aluminum chloride catalyst is not sensitive to air and moisture upon storage and much easier to handle than its soluble counterpart, AlCl₃. In addition, the catalyst was not influenced by water formed and not altered during the

TABLE 1 Solvent Effect on the Reaction of Aniline (1 mmol) with 2,5-Hexandione (1 mmol) Catalyzed by $PS/AICl_3$ (15 mol%)^a

Entry	Solvent	Conditions	Time (h)	Yield (%)
1	THF	Reflux	8	0
2	CHCl ₃	Reflux	8	Trace
3	CH ₂ Cl ₂	Reflux	8	Trace
4		Reflux	8	Trace
5	CH₃ŪH₂ŌH	Reflux	6	46
6	$CH_3CN(15 mol\%)^b$	Reflux	75 min	90
7	CH ₃ CN(20 mol%)	Reflux	75 min	90
8	Toluene	Reflux	8	Trace
9	Neat	80°C	8	Trace
10	Neat	100°C	8	Trace
11	CH₃CN ^c	Reflux	12	0

^aThe yields refer to the isolated pure products.

^bThe catalyst loading.

^cThe reaction was carried out in the absence of PS/AICl₃.

 TABLE 2
 Preparation of Pyrroles Using PS/AICl₃^a as Catalyst

Entry	Amine (1)	Product (3)	Time (min)	Yield ^b (%)	
1	C ₆ H₅NH₂	3a	75	90 ^{90,90,90,88,88,86,86^c}	
2	p-(CH ₃)C ₆ H ₄ NH ₂	3b	75	90	
3	O-(CH3)C6H4NH2	3c	70	90	
4	p -(OCH_3) $C_6H_4NH_2$	3d	55	92	
5	$p-(NO_2)C_6H_4)NH_2$	3e	135	83	
6	$p-(OH)C_6H_4NH_2$	3f	50	93	
7	p-(CI)C ₆ H ₄ NH ₂	3g	70	87	
8	C ₆ H ₅ CH ₂ NH ₂	3h	30	94	
9	p-(OCH ₃)C ₆ H ₄ CH ₂ NH ₂	3i	35	94	
10	CH ₃ CH ₂ CH ₂ NH ₂	3j	30	95	
11	CH ₃ (CH ₂) ₃ NH ₂	3k	40	93	
12	NH ₂	SI CH ₃ H ₃ C	35	94	
		$N - (CH_2)_2 N$			
13	NH ₂ (CH ₂) ₂ NH ₂	$3n \qquad CH_3 \qquad H_3C$	45	95	
		$CH_3 H_3C$ N-(CH_2) ₄ N			
14	$NH_2(CH_2)_4NH_2$	30 CH ₃ H ₃ C	45	94	

^aReaction conditions: amine (1 mmol); 2,5-hexandione (1 mmol); PS/AICl₃ (15 mol%); CH₃CN as solvent (10 mL); reflux condition. ^bIsolated yield.

^cReusability of catalyst after 8 times.

reaction, confirmed by the reusability and leaching test of PS/AlCl₃. To find out whether the reaction takes place in the solid matrix of PS/AlCl₃ or whether released AlCl₃ is responsible for the condensation reaction, PS/AlCl₃ was added to CH₃CN and the mixture was stirred at reflux temperature for 3 h. Then, the catalyst was filtered off and the filtrate was analyzed for its aluminum content, which showed a negligible release of AlCl₃. The filtrate was found to be inactive for the condensation reaction. This observation indicates that PS/AlCl₃ is stable under the reaction conditions, and there is no leaching of Lewis acid moieties during reactions.

With these results in hand, other amines (aliphatic and aromatic) have been subjected to the above-mentioned optimized conditions, and the results are listed in Table 2. It is evident that our methodology is reasonably general and can be applied to several amines. As indicated in Table 2, in all cases the reaction gives the corresponding pyrroles in good to excellent yields and completed within 30–135 min under reflux conditions. It is well known that the reactivity of aliphatic amines is more potent compared with that of aromatic amines, thus aliphatic amines give higher yields or shorter reaction times that are generally observed in other systems [26], and both lead to high yields of the

pyrrole products. As shown in Table 2, a series of aromatic amines bearing either electron-donating (Table 2, entries 2-4,6) or electron-withdrawing (Table 2, entries 5,7) groups on an aromatic ring was investigated and the results show that they are both effective in the Paal-Knorr condensation reaction. Moreover, we also examined the reaction of aliphatic and cyclic aliphatic amines with 2 (Table 2, entries 8–11,12). Similarly, the corresponding pyrrole products were obtained with excellent yields. In continuation of this study, we replaced the monoamino instead of diamino substrates in the same conditions in giving bipyrrole compounds (Table 2, entries 13,14) in excellent yields. In this reaction, 2 equiv of 2 were required to have a complete conversion of diamines.

The stability of the catalyst and its reusability are the main advantages of this polymeric catalyst. Therefore, the recovery and reusability of $PS/AlCl_3$ was examined. The catalyst can be separated and reused after washing with CHCl₃ and drying at 60°C. The reusability of the catalyst was investigated in the reaction of aniline with 2,5-hexandione (Table 2, entry 1) in the presence of 15 mol% PS/AlCl₃. The results illustrated in Table 2 showed that the catalyst can be used eight times without any loss of its activity. PS/AlCl₃ could be stored and handled under open-air conditions.

Entry	Catalyst	Conditions	Time (min)	Yield (%) ^a	Reference
1	CuCl ^b	Solvent-free	25	55	[28]
2	Cu(OTf) ₂	Solvent-free	25	78	i28i
3	Mg(OTf)2	Solvent-free	25	47	i28i
4	Bi(OTf)3	Solvent-free	25	72	[28]
5	Sm(OTf) ₃	Solvent-free	25	88	[28]
6	Eu(OTf) ₃	Solvent-free	25	88	i28i
7	Sc(OTf) ₃	CH ₂ Cl ₂	25	77	i28j
8	ZnCl	Solvent-free	1.5 h	51	[27]
9	CoCl	Solvent-free	2 h	47	27
10	NiCl2	Solvent-free	3 h	39	[27]
11	In(OTĪ) ₃	CH ₂ Cl ₂	1 h	91	27
12	InBr ₃	Solvent-free	1 h	84	[27]
13	Bi(NO ₃) ₃ .5H ₂ O	CH ₂ Cl ₂ /r.t.	10 h	96	[8]
14	`PS/AĬCI₃¯	CH ₃ ČN	75	90	Table 2

TABLE 3	Synthesis of P	vrrole 3a by	/ Reacting	Aniline	1a with 2	2.5-Hexandione	2 under	Various	Lewis Acid	d Cataly	vsts
			ricacting	, , , , , , , , , , , , , , , , , , , ,		_,0 110/01/01/01/01		vanous	LCW13 / 1010	Joalar	1010

^alsolated yield.

^b40 mol%.

The actual mechanism of the reaction is unclear. However, based on the experimental results and by referring to the literature [8,27,28], the mechanism of the pyrrole formation proceeds by the usual pathway proposed using Lewis acids.

Table 3 compares efficiency of $PS/AlCl_3$ (time, yield, reaction conditions) with efficiency of other Lewis acid catalysts in synthesis of **3a** obtained by other groups.

In conclusion, $PS/AlCl_3$ was found to be an efficient, environmentally benign, and stable heterogeneous polymer-supported Lewis acid catalyst for the preparation of N-substituted pyrroles. The mild reaction conditions, high to excellent yields, short reaction times, simple experimental procedure and product isolation, recyclability of the catalyst with no loss in its activity, low cost, and easy handling of the polymeric catalyst are important features of this new protocol to prepare pyrroles. In addition, unlike some other methods, no extra energy source such as microwave irradiation or ultrasound is needed for the success of the reaction.

EXPERIMENTAL

Materials and Instruments

All chemical reagents and solvents were obtained from Fluka (Switzerland) and Merck (Germany) chemical companies and were used without further purification. Cross-linked polystyrene (8% divinylbenzene) was prepared via suspension polymerization. PS/AlCl₃ was prepared using cross-linked polystyrene as reported in the literature [21b]. The capacity of the catalyst was determined by the Mohr titration method. All products were known compounds and were identified by comparison of their physical and spectral data with those of the authentic samples. Melting points were measured on an Electrothermal 535 apparatus and were uncorrected. ¹H NMR spectra were recorded on a Bruker DPX-250 Avance spectrometer at 250.13 MHz. IR spectra were recorded on a Unicam Matteson 1000 spectrophotometer. Reaction monitoring and purity determination of the products were accomplished by TLC on silicagel polygram SILG/UV₂₅₄ plates. All yields refer to isolated products.

Preparation of PS/AlCl₃

Anhydrous AlCl₃ (4.5 g) was added to polystyrene (8% divinylbenzene, 3.5 g) in carbon disulfide (25 mL) as the reaction medium. The mixture was stirred using a magnetic stirrer under reflux condition for 50 min, cooled and then water (40 mL) was cautiously added to hydrolyze the excess AlCl₃. The mixture was stirred until the deep orange color disappeared and the polymer became light yellow. The polymer beads were then filtered and washed with water (300 mL) and then with acetone and diethyl ether. The polymer was dried in a vacuum oven for 12 h at room temperature. The capacity of the polymeric catalyst based on its chloride content was 0.47 mmol AlCl₃/g catalyst [29].

General Experimental Procedure for the Synthesis of Pyrroles 3:

In a round-bottom flask (50 mL) equipped with a condenser and a magnetic stirrer, a solution of the amine (1 mmol) and 2,5-hexandione (1 mmol) in CH₃CN (10 mL) was prepared. PS/AlCl₃ (15 mol%, 0.15 mmol) was added to the solution, and the reaction mixture was stirred magnetically under reflux conditions for the given time (Table 2). The progress

of the reaction was monitored by TLC. After completion of the reaction, the catalyst was filtered off and washed with CHCl₃ (2×10 mL) to remove any adhering pyrrole product and the filtrate concentrated on a rotary evaporator under reduced pressure to give the crude product. The residue was purified by column chromatography and by recrystallization to afford the pure pyrrole derivatives. The spent polymeric catalyst from different experiments was combined, washed with CHCl₃ and dried overnight in a vacuum oven and reused. Representative examples of spectroscopic and analytical data are given below.

Compound **3a**: mp = 50°C; ν_{max}/cm^{-1} 3059, 2921, 1600, 1520, 1497, 1403, 1320; $\delta_{H}(250 \text{ MHz, CDCl}_{3})$ 2.05 (s, 6H, C<u>H</u>₃), 5.92 (s, 2H, pyrrole ring), 7.22–7.26 (2H, m, Ar–H), 7.42–7.50 (3H, m, Ar–H). Found: C, 79.89; H, 7.48; N, 8.00. C₁₂H₁₃N requires C, 84.16; H, 7.65; N, 8.18%.

Compound **3h**: Oily material; ν_{max}/cm^{-1} 3061, 2923, 2845, 1560, 1520, 1495, 1401, 1320; δ_{H} (250 MHz, CDCl₃) 2.17 (6H, s, C<u>H</u>₃), 5.01(2H, s, CH₂), 5.91 (2H, s, pyrrole ring), 6.90–6.93 (2H, m, Ar–H), 7.21–7.36 (3H, m, Ar–H). [Found: C, 84.36; H, 8.19; N, 7.53. C₁₃H₁₅N requires C, 84.39; H, 8.16; N, 7.56].

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