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# Phosphorus, Sulfur, and Silicon and the Related Elements

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# Functionalized Poly(Amidoamine) Dendrimer as a Strong Ionic Brønsted Acid Organocatalyst for Protection/ Deprotection of Aldehydes

Ali Pourjavadi<sup>a</sup> & Seyed Hassan Hosseini<sup>a</sup>

<sup>a</sup> Polymer Research Laboratory, Department of Chemistry, Sharif University of Technology, Tehran, Iran Accepted author version posted online: 07 Oct 2014.Published online: 13 Dec 2014.

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## FUNCTIONALIZED POLY(AMIDOAMINE) DENDRIMER AS A STRONG IONIC BRØNSTED ACID ORGANOCATALYST FOR PROTECTION/DEPROTECTION OF ALDEHYDES

## Ali Pourjavadi and Seyed Hassan Hosseini

Polymer Research Laboratory, Department of Chemistry, Sharif University of Technology, Tehran, Iran

#### **GRAPHICAL ABSTRACT**



Abstract Poly(amidoamine) dendrimer (PAMAM) was successfully functionalized by chlorosulfonic acid to form an strong ionic acid catalyst. The resulting polymeric catalyst was shown to be an efficient catalyst for the synthesis of 1,1-diacetyl from aldehydes under free solvent conditions at room temperature. Also, the deprotection of the resulting aldehydes was investigated under catalytic conditions. The Hammett acidity function of catalyst showed that catalyst is a strong Brønsted acid. Due to the multi-functional nature of PAMAM, the catalyst has high loading level of acidic protons.

Keywords Poly(amidoamine); organocatalyst; polymeric catalyst; 1,1-diacetyl

#### INTRODUCTION

Molecular catalysis is still an active research area in both academia and industry because it is interesting in the green chemistry concept.<sup>1</sup> These molecular catalysts are widely used in the production of fine chemicals in the drug industry and agriculture. Solution-based methodologies rely not only on the optimization and discovery of molecular catalysts for specific transformation but also separation of catalyst from reaction products.<sup>2</sup> Although, using heterogeneous catalysts such as supported silica or metal oxides seems to be good for industry or the academy, there are several drawbacks to using heterogeneous catalysts that generally limit their widespread application to in batch processes. These drawbacks include difficulty in the synthesis of immobilized heterogeneous catalysts, nonuniformity in catalyst structure, slow diffusion of substrates therein, high catalyst leaching and lower

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Address correspondence to Ali Pourjavadi, Polymer Research Laboratory, Department of Chemistry, Sharif University of Technology, Tehran, Iran. E-mail: purjavad@sharif.edu

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overall activities than the homogeneous system.<sup>4</sup> To overcome these disadvantages, soluble polymers have been used for immobilization of catalysts.<sup>5</sup> In this manner, the desirable features of homogeneous catalysis, such as comparable reaction kinetics and mass transfer, can be maintained while the macromolecular nature of the polymer provides a convenient means of purification and, in some cases, recyclability.<sup>2</sup>

Both linear and branched polymers have been used as soluble macromolecular supports for reagents and catalysts.<sup>6</sup> While both polymer species are, in general, readily available, linear polymers can suffer from poor loading capacity, because only two or one side (head and tail) of linear polymers can be grafted by reagents or catalysts. For example, MeO-PEG<sub>5000</sub> catalyst conjugates carry a loading of only 0.2 mmol catalyst per gram of polymer, while branched polymers typically carry several mmol.g<sup>-1</sup> of catalysts.<sup>7</sup> Many papers have described both the covalent and noncovalent grafting of reagent or catalysts into the branched polymers.<sup>8</sup> But as far as we are aware, there is no report about the synthesis of a Brønsted acid functionalized PAMAM dendrimer.

Herein, we report the synthesis of PAMAM functionalized sulfamic acid. Addition to the acidic properties of the dendrimer catalyst, the catalyst has ionic structure. The resulting catalyst was used for the protection of aldehydes in 1,1-diacetate form. Protection of aldehydes as acylals is often preferred due to their ease of preparation and their stability toward basic and neutral conditions.<sup>9</sup> Generally, they have synthesized from acetic anhydride and aldehydes using strong protonic acids<sup>10</sup> and Lewis acids.<sup>11</sup> But these methods suffer from some drawbacks such as high catalyst loading, metal contain catalysts, and using hazardous organic solvent.

#### **RESULT AND DISCUSSION**

#### Synthesis of Catalyst

We chose PAMAM dendrimer as a source of proton-affinity bases and multifunctional polymer for the formation of the Brønsted acid catalyst. It is well-known that PAMAM dendrimers are monodisperse, hyperbranched polymers possessing a very high concentration of primary and tertiary amine groups, which can be readily protonated to form cationic centers<sup>3</sup>.

The first generation of PAMAM dendrimer (G1) was prepared according to the standard procedure.<sup>12</sup> For sulfamic acid functionalization, chlorosulfonic acid was added to a solution of PAMAM in dry dichloromethane. The synthetic protocol is summarized in Scheme 1.The terminal amine groups of PAMAM react with ClSO<sub>3</sub>H to form sulfamic acid functionalities. The tertiary inner amine groups in PAMAM structure neutralize by ClSO<sub>3</sub>H. After washing with water, ClSO<sub>3</sub><sup>-</sup> counterions react with H<sub>2</sub>O molecules to obtain HSO<sub>4</sub> as counterions.

The FT-IR spectra of PAMAM before and after functionalization are presented in Figure S1 (Supplemental Materials). The FT-IR spectrum of PAMAM shows a strong peak at 1663 cm<sup>-1</sup> corresponding to the amide groups in the dendrimer. After functionalization of PAMAM with chlorosulfonic acid, a strong peak at 1083 cm<sup>-1</sup> and 1377, corresponding to sulfate anions and sulfamic groups, appeared. The same carbonyl peaks can be seen at the spectrum of functionalized PAMAM, which shows the amide groups of dendrimer remain intact during the functionalization.

Figure S2 shows the thermal gravimetric analysis (TGA) curves of PAMAM (G1) and PAMAM@SO<sub>3</sub>H. As seen, the decomposition of PAMAM dendrimer initiates at 190°C,



Scheme 1 Synthesis of PAMAM-sulfamic acid functionalized dendrimer.

whereas PAMAM@SO<sub>3</sub>H is decomposed up to  $240^{\circ}$ C. It is useful for catalytic applications that the catalyst be stable at higher temperature.

Figure S3 shows the <sup>1</sup>H NMR of catalyst. Because of positive N atoms in the structure of catalyst, all peaks are shifted to higher values<sup>12</sup>.

The elemental analysis of catalyst is shown in Table S1. Comparison between theoretical and experimental CHNS analysis shows same the content of each element. These results demonstrate that proposed structure of catalyst is valid.

We calculated the acidity of catalyst by determination of the Hammett acidity function. The Hammett acidity function expresses the acidity strength of an acid.<sup>13</sup> It can be calculated by the equation below:

$$H_0 = pK(In)_{aq} + \log([In]_s/[HIn]_s)$$

Where, [In] and [HIn] are concentration of basic indicator. The value of  $[In]_s/[HIn]_s$  was determined through UV-visible spectrum. As seen in Table 1, concentrated sulfuric acid as a strong acid shows  $H_0 = 1.38$ . Sulfamic acid and methyl sulfamic acid with a weaker acidity than sulfuric acid have  $H_0 = 1.60$  and 1.63, respectively. The acidity function for PAMAM@SO<sub>3</sub>H shows that acidity of PAMAM@SO<sub>3</sub>H is more than NH<sub>2</sub>SO<sub>3</sub>H and

Entry	Catalyst	A <sub>max</sub>	[I](%)	[HI](%)	$H_0$
1	_	1.34	100	0	_
2	$H_2SO_4$	0.94	71.22	28.78	1.38
3	NH <sub>2</sub> SO <sub>3</sub> H	1.03	80.40	19.60	1.60
4	CH <sub>3</sub> NHSO <sub>3</sub> H	1.01	81.32	18.68	1.63
5	PAMAM@SO3H	0.98	72.41	27.59	1.41

Table 1 Determination of acidity by Hammett acidity functions

Condition for Hammett acidity function: solvent: water, indicator: 4-nitroaniline (p*K*(I)aq = 0.99),  $5.0 \times 10^{-4}$  mol/L, Catalyst:  $7.5 \times 10^{-3}$  mol/L, 27°C.

CH<sub>3</sub>NHSO<sub>3</sub>H. The stronger acidity of PAMAM@SO<sub>3</sub>H can be attributed to the ionic nature of dendrimeric acid.

The activity of catalyst was tested in the synthesis of 1,1-diacetyl using the reaction between aldehydes and acetic anhydride at room temperature (Scheme 2). We have also investigated the deprotection of acylals in methanol medium using PAMAM@SO<sub>3</sub>H as a catalyst. It was postulated that, the same catalyst in the presence of nucleophile (MeOH) would bring about deacylation via protonation of carbonyl carbon in acylals.



Scheme 2 Acetylation of aldehydes using Ac<sub>2</sub>O and their deprotection in the presence of PAMAM@SO<sub>3</sub>H.

Table 2 shows the control experiment and optimization of the catalytic synthesis of 1,1-diacetyl by choosing the reaction between benzaldehyde and acetic anhydride as a model reaction. No product was separated in the absence of catalyst. When 10 mol% of concentrated sulfuric acid was used as the catalyst, 92% yield was obtained in 15 minutes. Using 51 mol% of sulfamic acid as the catalyst also gave 92% yield in 1 hour at room temperature. Optimization of the catalyst amount shows 0.4 mol% of PAMAM@SO<sub>3</sub>H is

Table 2 Control experiments of attempted diacetylation reaction

Entry	Catalyst	Amount of catalyst (mol%)	Solvent	Time (min)	Vield (%) <sup>a,b</sup>	
	Cuturyst	Thiodile of Calaryst (morito)	Borrein	Time (iiiii)		
1		_	_	24 h	14	
2	$H_2SO_4$	10	_	15	92	
3	NH <sub>2</sub> SO <sub>3</sub> H	51	_	60	92	
4	PAMAM@SO3H	0.8	_	30	93	
5	PAMAM@SO3H	0.4	_	30	91	
6	PAMAM@SO3H	0.2	_	30	72	
7	PAMAM@SO3H	0.4	$CH_2Cl_2$	30	77	
8	PAMAM@SO3H	0.4	CH <sub>3</sub> CN	30	86	
9	PAMAM@SO3H	0.4	$Et_2O$	30	62	
10	PAMAM@SO3H	0.4	THF	30	75	
11	PAMAM@SO3H	0.4	Hexane	30	42	

<sup>a</sup> Reaction condition; benzaldehyde (1 mmol), acetic anhydride (5 mmol), at room temperature. <sup>b</sup>GC yield.

Entry	Ar	Protection <sup>a</sup>		Deprotection <sup>b</sup>		
		Time (min)	Yield (%)	Time (min)	Yield (%)	Ref.
1	Ph	30	91	35	94	10
2	4-(Cl)Ph	20	94	20	90	10
3	4-(Br)Ph	20	95	20	93	16
4	4-(Me)Ph	30	84	25	91	18
5	4-(HO)Ph	60	89	40	90	16
6	2-(HO)Ph	60	92	20	93	10
7	4-(MeO)Ph	45	92	20	90	18
8	2-(MeO)Ph	120	89	20	89	18
9	4-(NO2)Ph	20	91	35	95	10
10	3-(NO2)Ph	20	94	30	94	10
11	2-(NO <sub>2</sub> )Ph	60	86	30	95	10
12	4-(CO <sub>2</sub> H)Ph	20	94	15	89	21
13	4-(CN)Ph	20	90	20	93	21
14	Vanillin	60	88	40	93	10
15	Furfural	15	92	15	92	10
16	4-(CHO)Ph <sup>c</sup>	120	87	60	93	14

Table 3 Acetylation of aldehydes using  $Ac_2O$  and their deprotection in the presence of PAMAM@SO<sub>3</sub>H at room temperature

<sup>a</sup> Reaction condition: Aldehyde (1 mmol), Ac<sub>2</sub>O (5 mmol), Catalyst (0.4 mol%), solvent free, room temperature, isolated yields.

<sup>b</sup> Reaction condition: Acylal (0.5 mmol), Methanol (2 mL), Catalyst (0.4 mol%), room temperature, isolated yields.

<sup>c</sup> Both of aldehyde groups were converted to acylal and both were deprotected.

sufficient for the completion of reaction at room temperature. The same reactions were performed in various solvents, but solvent-free conditions gave the best yield.

Various aldehydes include hindered aldehydes and highly deactivated aldehydes were also diacetylated with PAMAM@SO<sub>3</sub>H in excellent yield (Table 3). Also, the resulting acylals were converted to the corresponding aldehydes in the presence of PAMAM@SO<sub>3</sub>H in excellent yield and the results are summarized in Table 3.

We also investigated the chemoselective protection of aldehydes in the presence of ketones. As shown in Scheme 3, 1:1 mixture of benzaldehyde and acetophenone in the presence of the catalyst gave only the aldehyde diacetyl.



Scheme 3 Chemoselective acetylation of aldehydes in the presence of ketones.

Table 4 shows a comparison between various catalytic systems which have reported the synthesis of 1,1-diacylal of benzaldehyde. However, the yield of reaction is excellent for all of the catalytic systems (based on GC yield) but they suffer from some drawbacks such

Entry	Catalyst	Reaction condition	Yield (%)	Ref.
1	NH <sub>2</sub> SO <sub>3</sub> H (40 mol%)	Solvent free, 60 min, r.t.	90	10
2	NH2SO3H (40 mol%)	Solvent free, 10 min, r.t. ultrasound	94	10
3	Cu(BF <sub>4</sub> ) (1mol%)	Solvent free, 20 min, r.t.	96	11
4	PEG-SO <sub>3</sub> H(1.67mol%)	CH <sub>2</sub> Cl <sub>2</sub> , 18 min, r.t	97	14
5	SiO <sub>2</sub> @HClO <sub>4</sub> (0.5 mol%)	Solvent free, 2 min, r.t.	97	15
6	Cu(ClO <sub>4</sub> ) <sub>2</sub> (0.05mol%)	Solvent free, 1 min, r.t.	98	16
7	LiOTf (20 mol%)	Solvent free, 12 h, r.t.	96	17
8	H <sub>3</sub> PW <sub>12</sub> O <sub>40</sub> (2 mol%)	Solvent free, 35min, r.t.	99	18
9	Amberlyst-15 (0.5g)	CH <sub>2</sub> Cl <sub>2</sub> , 45 min, r.t	95	19
10	Fe <sup>3+</sup> -Montmorillonite (50mg)	Solvent free, 10 min, r.t.	98	20
11	Fe <sub>3</sub> O <sub>4</sub> @Poly(ionic liquid) (50mg)	Solvent free, 20 min, r.t.	91	21
12	[HSO <sub>3</sub> -pmim][CH <sub>3</sub> SO <sub>3</sub> ] (10mol%)	Solvent free, 10 min, r.t. ultrasound	97	22
13	PAMAM@SO <sub>3</sub> H (0.4mol%)	Solvent free, 30 min, r.t.	91	This work

Table 4 Comparison between activity of various catalysts in the synthesis of 1,1-diacylal of benzaldehyde

as high catalyst loading (entries 1,2,7,10,11,12), metal contain catalysts (3,6,7,8,10,11), and using organic solvents (4,9). The multi-acidic functionalities of catalyst lead to using low amount of PAMAM@SO<sub>3</sub>H for the reaction. Also, the ionic nature of the catalyst can promote the acidity and activity of the acidic catalyst. Due to polymeric nature of PA-MAM, the separation of catalyst is easy. Also, PAMAM@SO<sub>3</sub>H has large amount of acidic protons which is useful in the large-scale application when catalyst is used in low weight percent.

The recyclability of catalyst was also investigated and the results show that no significant loss of catalyst activity was observed (Figure 1). Due to the polymeric structure of catalyst, the separation of catalyst is easier than for homogenous catalysts.



Figure 1 Recyclability of catalyst in the synthesis of 1,1-diacylal of benzaldehyde.

#### **EXPERIMENTAL**

#### General Procedure for Protection and Deprotection of Aldehydes

To a magnetically stirred solution of aldehyde (1 mmol) and freshly distilled acetic anhydride (5 mmol), PAMAM@SO<sub>3</sub>H (10 mg, 0.4 mol%) was added at room temperature, and the mixture was stirred until complete disappearance of aldehydes (as monitored by TLC). After completion of reaction,  $CH_2Cl_2$  was added and the catalyst was extracted and dried at 50°C for another run. The organic layer was washed with saturated NaHCO<sub>3</sub> (3 × 25 mL) and water (15 mL), and dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>. Evaporation of the solvent under reduced pressure and recrystallization from ethanol gave the almost pure 1,1-diacetate.

For deprotection of corresponding 1,1 diacetates 0.5 mmol of prepared acylal and PAMAM@SO<sub>3</sub>H (5 mg, 0.4 mol%) was stirred in methanol (2 mL) at room temperature. Solvent was evaporated and catalyst was extracted by  $CH_2Cl_2$ . The organic layer was washed with saturated NaHCO<sub>3</sub> (3 × 25 mL) and water (15 mL), and dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>. Evaporation of the solvent under reduced pressure and recrystallization from ethanol gave the almost pure aldehyde.

#### CONCLUSION

We have synthesized a green catalyst which PAMAM dendrimer is functionalized by chlorosulfonic acid to and forms a strong acidic catalyst. The resulting catalyst was used in a green condition for protection and deprotection of aldehydes. The catalyst has a high capacity of protons and could be used in the low weight percent compared with substrates. Because of the macromolecular nature of the catalyst, separation of catalyst from the reaction medium is easy. In the view of green chemistry, the preset protocol is useful for large-scale production.

#### SUPPLEMENTAL MATERIAL

Supplementary data of this article can be accessed on the publisher's website, www.tandfonline.com/gpss

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