

Polymer-supported sulfonimide as a novel water-tolerant Brønsted acid catalyst for esterification of equimolar carboxylic acids and alcohols

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Abstract

A novel polymer-supported sulfonimide (PS-SI), 10-(4'-perfluorobutylsulfonamino sulfonylphenyl)decylpolystyrene, was synthesized and characterized by FT-IR, solid state ^{19}F NMR, SEM and TGA. The acid strength determination indicated that PS-SI was a strongly acidic polymer. The catalytic performance of the polymer catalyst was tested by direct esterification of equimolar carboxylic acids and alcohols in excess water. The results showed that PS-SI was an effective Brønsted-acid catalyst and could be reused several times without any loss of catalytic activity.

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Keywords: Polymer-supported; Solid Brønsted acid; Perfluorobutylsulfonimide; Water tolerant catalyst; Esterification

1. Introduction

Organic reactions in aqueous media have attracted great attention of chemists in recent years [1]. The use of aqueous solution of reactants has many advantages; it is economical, nontoxic, safe, and environment friendly and makes it unnecessary to dry the reagents. Therefore various organic reactions have been developed in aqueous media [2], but there are still some reactions which are difficult to carry out in the presence of excess water, the direct esterification of carboxylic acids with alcohols being one of such examples.

Esterification is an important acid-catalyzed reaction in organic synthesis. Because water impedes the equilibrium shift to the ester formation, the direct esterification of equimolar carboxylic acids with alcohols in water is hard to carry out. Recently, some efficient systems of esterification in excess water have been reported [3–6]. Among various catalysts the polymer-supported Brønsted acids were found to be an important water-tolerant catalysts. But till now, most of the researches were mainly focused on sulfonic acid functionalized polymers [5,7,8]. Because of the high acid strength [9], the polymers containing sulfonimide groups or sulfon-

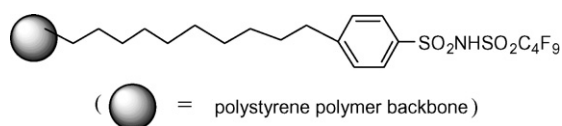
imide groups have recently received considerable attention in the development of new polymer-supported Brønsted acids [10–12]. Based on our previous work [13], in this paper we incorporated a long chain alkylphenyl group into polystyrene resin to develop a new polymer-supported sulfonimide catalyst (PS-SI), 10-(4'-perfluorobutylsulfonamino sulfonylphenyl)decylpolystyrene (Scheme 1), which showed high catalytic activity for the condensation of equimolar carboxylic acids and alcohols in water.

2. Experimental

All reagents (A.R.) were purchased from China National Pharmaceutical Group Corporation (Shanghai) and treated according to standard methods. The liner polystyrene beads were purchased from Aldrich and used as received. NKC-9 was purchased from Nakai Chemical Plant and Nafion 50 was obtained from Wako Pure Chemical Industries Ltd. (Japan).

The NMR experiment was carried out at 9.4 T on a Varian Infinityplus-400 spectrometer with use of a double-resonance 4-mm probe capable of a maximum sample spinning speed of 16 kHz. The resonance frequency for ^1H and ^{19}F are 400.12, 376.45 MHz, respectively. The ^{19}F MAS NMR spectra were acquired using spin echo with a ^{19}F pulse width ($\pi/2$) of 4.0 μs and an echo delay of 285 μs . The rotor end caps and spacers were made of polypropylene to minimize ^{19}F background

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Scheme 1. Polymer-supported sulfonimide catalyst (PS-SI), 10-(4'-perfluorobutylsulfonaminosulfonylphenyl)decylpolystyrene.

signal. Acquisition delay of 10 s was estimated to be sufficient for obtaining quantitative spectra. All spectral chemical shifts were referenced to CFCl_3 .

IR spectra were recorded on a Bruker Vertex 70 Fourier Transform instrument (the variable temperature cell P/N 20610 solids Holder, 50–200 °C). TG and SEM analysis were performed on a Perkin-Elmer TG97 thermal analysis instrument (heating rate of 10 °C/min) and on a Quanta 200 microscope (the samples were coated with gold powder on 682 Gatan Inc.), respectively. GC analysis was carried out using a FuLi 9790 chromatograph equipped with a flame ionization detector.

2.1. Synthesis of 10-(4'-perfluorobutylsulfonaminosulfonylphenyl)decylpolystyrene (PS-SI)

The synthesis route of PS-SI is shown in Scheme 2 [14–19].

2.1.1. Synthesis of $\text{C}_4\text{F}_9\text{SO}_2\text{NH}_2$

$\text{C}_4\text{F}_9\text{SO}_2\text{NH}_2$ was prepared according to the literature [14,15]. IR (KBr, ν , cm^{-1}): 3346, 3182 (N–H); 1380, 1139 ($-\text{SO}_2-$); 1199 (C–F).

2.1.2. Synthesis of 10-benzene-9-ketone acid (**1**)

Mono methyl sebacate 13.0 g (0.057 mol) and SOCl_2 12 mL (0.165 mol) were added to a one-neck flask and stirred at 50 °C for 6 h. The mixture was distilled and 11.7 g of mono methyl

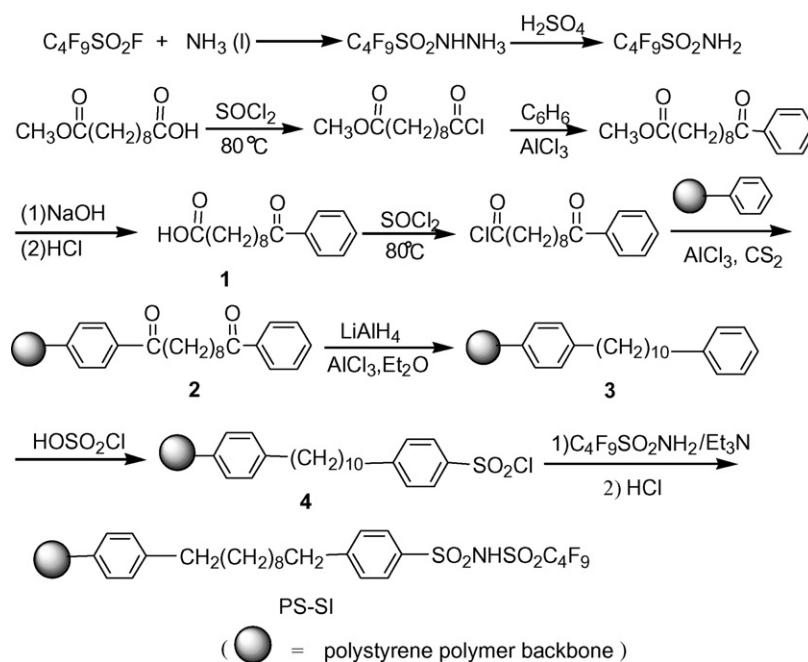
sebacoyl chloride was obtained (148–150 °C, 4 mmHg, 90% yield).

To a mixture of AlCl_3 (10 g) and newly refined benzene (20 mL) in a three-neck flask, mono methyl sebacoyl chloride was added dropwise at room temperature, and stirred at 80 °C for 6 h. After the mixture was cooled to room temperature, 15% hydrochloric acid solution (40 mL) was added slowly. The organic layer was washed with saturated NaHCO_3 solution and distilled water twice respectively. The excess benzene was distilled off and 8.9 g (ca. 60% yield) of gross product mono methyl 10-benzene-9-ketone acid was obtained. Without further treatment, the gross product was dissolved in 4 mL alcohol, 15% sodium hydroxide solution (20 mL) was added and the mixture was stirred at 100 °C for 5 h. The mixture was then transferred to a 200 mL beaker immediately and 15% hydrochloric acid solution (30 mL) was added. The obtained buff solid was filtered and washed with distilled water till the filtrate was neutral and dried under vacuum at 60 °C. After recrystallization with petroleum ether (60–90 °C), 7.0 g of 10-benzene-9-ketone acid (**1**) was obtained as white solid, mp 82 °C. IR (KBr, ν , cm^{-1}): 2930, 2850 ($-\text{CH}_2-$); 3058, 1596, 1466 ($-\text{C}_6\text{H}_5$); 1710 ($-\text{COOH}$); 1685 ($-\text{CO}-\text{Ph}$).

2.1.3. Acylation of polystyrene

1 (4.0 g, 0.015 mol) and SOCl_2 (10 mL, 0.137 mol) were added to a one-neck flask and stirred at 50 °C for 6 h. The excess SOCl_2 was removed by distillation and the remainder 10-benzene-9-ketone acid chloride was used for the next step synthesis without further treatment.

10-Benzene-9-ketone acid chloride was dissolved in 10 mL CS_2 , added dropwise to the solution of polystyrene beads (2.5 g) and trituated AlCl_3 (2.0 g) dispersed in 40 mL CS_2 . The mixture was stirred at 50 °C for 24 h, and then 5% hydrochloric acid solution (100 mL) was added slowly. The obtained buff solid



Scheme 2. Synthesis route of PS-SI.

was washed with H₂O, H₂O–THF (1:1) and THF successively, dried overnight under vacuum at 80 °C to give 2.89 g of polymer **2**. IR (KBr, ν , cm⁻¹): 2930, 2850 (–CH₂–); 3058, 1596, 1466 (–C₆H₅); 1678 (–CO–Ph).

2.1.4. Deoxidation of polymer 2

To 2.0 g of polymer **2** and 2.8 g of AlCl₃ dispersed in 40 mL of Et₂O at 0 °C in argon atmosphere, were added 0.8 g of LiAlH₄ carefully. The mixture was then stirred at 50 °C for 15 h. When it was cooled to 0 °C, 5% hydrochloric acid solution (120 mL) was added dropwise, and the mixture was kept stirring until the water phase became transparent. After filtration the polymer was washed successively with H₂O, H₂O–THF (1:1) and THF, and then dried overnight under vacuum at 80 °C to give 1.8 g of polymer **3**. IR (KBr, ν , cm⁻¹): 2930, 2850 (–CH₂–); 3058, 1596, 1466 (–C₆H₅);

2.1.5. Chlorosulfonation of polymer 3

ClSO₂OH (15 mL) was added dropwise to a stirred mixture of 1.6 g of **3** and 25 mL of ClCH₂CH₂Cl at 80 °C in argon atmosphere. The polymer obtained 2 h later was then filtered and washed successively with CHCl₃, THF, THF–water (1:1) and THF. After drying under vacuum at 80 °C for 6 h, the polymer was treated with excess amount of thionyl chloride and small amount of pyridine for 16 h at reflux temperature in argon atmosphere, then immediately poured into ice water. When the mixture was cooled to room temperature, the polymer was filtrated and washed with distilled water until the filtrate was neutral, and then dried under vacuum at 80 °C overnight to give 2.51 g of polymer **4** (loading of –SO₂Cl = 3.20 mmol/g, –SO₂Cl content was determined by argentometry). IR (KBr, ν , cm⁻¹): 2930, 2850 (–CH₂–); 3058, 1591, 1453 (–C₆H₅); 1374 (–SO₂Cl); 1170 (–S=O).

2.1.6. Sulfamidation of polymer 4

Triethylamine (5 mL, 0.033 mol) was careful dripped into the mixture of polymer **4** (2.0 g, loading of –SO₂Cl = 3.20 mmol/g, –SO₂Cl content was determined by argentometry) and 4.0 g of C₄F₉SO₂NH₂ (0.0134 mol) in 50 mL of 1,2-dichloroethane. The mixture was stirred at 60 °C for 48 h in argon atmosphere, and then cooled to room temperature. After filtration the polymer was washed with CHCl₃, dense hydrochloric acid and distilled water successively, and then dried under vacuum at 80 °C for 24 h to give 4.1 g of the target product PS-SI. IR (KBr, ν , cm⁻¹): 3174 (N–H); 1600, 1406 (–C₆H₅); 1178 (–SO₂–); 1218 (C–F).

2.2. Determination of acidic strength and content of PS-SI

The acidic strength of the PS-SI was measured by the Hammett indicator method according to the literature [13].

Acid content of the PS-SI was determined by acid–base titration. A suspension of the PS-SI was stirred in a standard sodium hydroxide solution. After 4 h, the polymer was filtered out and washed with distilled water repeatedly till the filtrate was neutral. The filtrate was titrated with a standard hydrochloric acid solution with 0.01% phenolphthalein in ethanol as indicator. The

content of the PS-SI was determined by several parallel experiments.

2.3. General procedures for esterification

A mixture of a carboxylic acid (1.26 mmol), an alcohol (1.26 mmol), catalyst PS-SI (0.05 equiv. to the carboxylic acid) and water (5.56 mmol) was stirred at 80 °C. After 48 h, 50 μ L of the mixture was taken out; the catalyst was filtrated and washed by 50 μ L ethanol. The filtrate was analysed by gas chromatography.

3. Results and discussion

3.1. Acid strength and content of PS-SI

The acid strength of PS-SI determined with the Hammett indicators is listed in Table 1, which shows that PS-SI was a strong acid polymer ($H_0 \sim -11.35$). The acid content of PS-SI was found to be 3.19 mmol/g by titration.

3.2. FT-IR analysis of PS-SI

Compared with the chlorosulfonyl polymer in Fig. 1(a), the characteristic absorption bands of –SO₂Cl (ν_{as} 1374 cm⁻¹, ν_s 1170 cm⁻¹) almost disappeared after sulfamidation (Fig. 1(b)). The peaks at 1591, 1453 and 1415 cm⁻¹ can be assigned to the skeletal vibration of benzene ring. The peak at 1218 cm⁻¹ was attributed to the stretching vibration of C–F. The weak peak of

Table 1
Acidic strength of the PS-SI

Catalysts	Indicator and H_0		
	Antraquinone, –8.2	<i>p</i> -Nitrotoluene, –11.35	4-Chloronitrobenzene, –12.70
PS-SI	+	±	–

(+) Colour changed clearly; (–) colour unchanged; (±) colour changed unclearly.

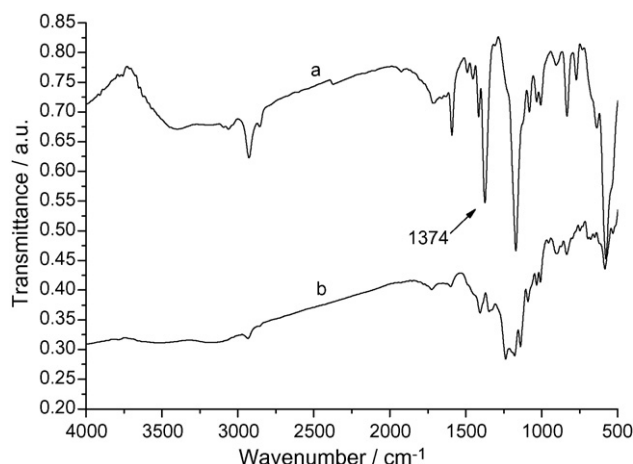


Fig. 1. FT-IR of chlorosulfonyl polymer (a) and PS-SI (b).

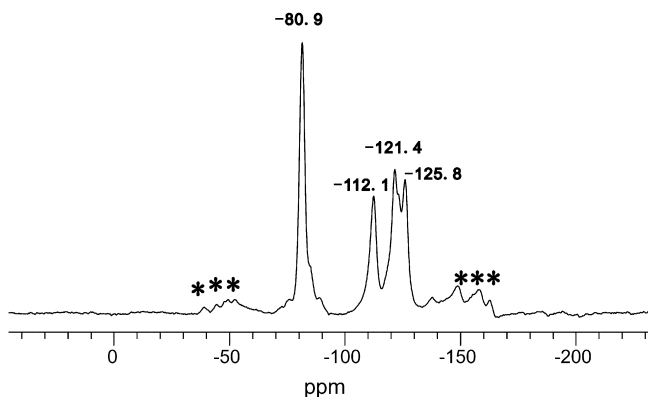


Fig. 2. Solid-state ^{19}F NMR spectrum of PS-SI.

3200 cm^{-1} in Fig. 1(b) can be assigned to the stretching vibration of N–H [20,21].

3.3. Solid-state ^{19}F NMR of PS-SI

The ^{19}F MAS NMR spectrum of PS-SI is shown in Fig. 2. The spectrum was obtained with 128 transients at room temperature and using 2048 points. It consisted of four well-resolved peaks at -80.9 , -112.1 , -121.4 and -125.8 ppm, respectively. The resonance at -80.9 ppm can be assigned to the $-\text{CF}_3$ at the end of the alkyl chain, and the peak at -112.1 ppm is attributed to the $-\text{CF}_2$ next to the $-\text{CF}_3$. According to the literature assignment [22,23], the peaks at -121.4 and -125.8 ppm can also be assigned to the $-\text{CF}_2\text{CF}_2\text{CF}_2\text{CF}_3$, and $-\text{CF}_2\text{CF}_2\text{CF}_2\text{CF}_3$, respectively. The integrated intensity ratio of each peak was around 3:2:2:2, which is consistent with the molecule formula. The peaks marked with an asterisk in Fig. 2 were assigned to the sidebands of the sample in the measurement. A combination of the assignment and the quantitative analysis can help us confirm the existence of $-\text{CF}_2\text{CF}_2\text{CF}_2\text{CF}_3$ group in the polymer catalyst.

3.4. SEM of PS-SI

The SEM images (Fig. 3) show that the polymer acid catalyst, PS-SI, was the irregular particles with an average size of

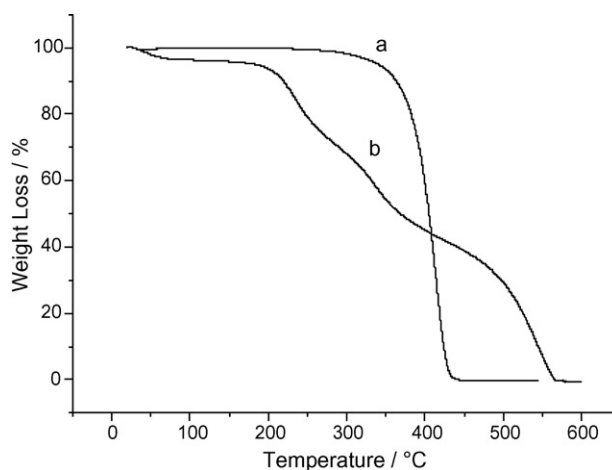


Fig. 4. Thermal analyses of samples: (a) TGA of polystyrene and (b) TGA of PS-SI.

about $10\text{--}20\ \mu\text{m}$ (Fig. 3(a)) and a rough surface (Fig. 3(b)). In the synthesis of PS-SI, the liner polystyrene used as polymer support was soluble in carbon disulfide but after acylation by 10-benzene-9-ketone acid chloride the polymer **2** was insoluble and it was deposited out from the reaction solution. So, the chemical method of preparation determined the irregular distribution and rough surface of PS-SI. The fairly high acid content of PS-SI (3.19 mmol/g) implies that most of the acid groups were on the surface of these particles.

3.5. Thermal analysis of PS-SI

The thermal analysis results are shown in Fig. 4. The TGA curve of polystyrene (Fig. 4(a)) shows that the polymer support was highly stable below $300\text{ }^\circ\text{C}$ and there are four main stages of weight loss for PS-SI (Fig. 4(b)). About 4% weight loss before $100\text{ }^\circ\text{C}$ could be attributed to the loss of the combined water or small organic molecules [24]. The pendant $\text{C}_4\text{F}_9\text{SO}_2\text{NH-}$ of PS-SI might began to decompose at $160\text{ }^\circ\text{C}$, resulting in about 25% weight loss in the temperature region of $160\text{--}250\text{ }^\circ\text{C}$. This result is consistent with the acid content by titration. The weight

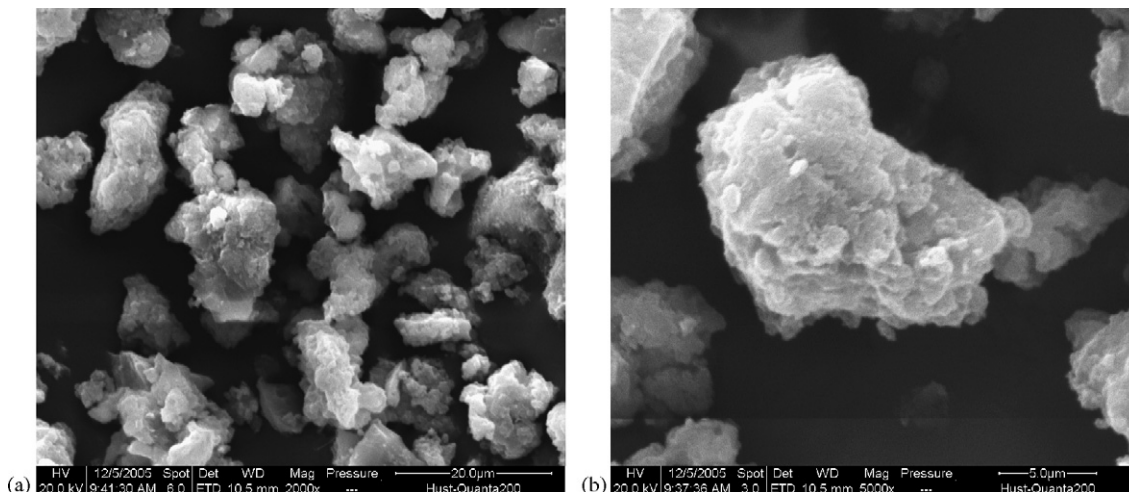


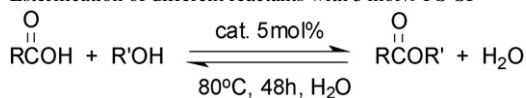
Fig. 3. SEM micrographs of PS-SI: (a) magnification of $2000\times$ and (b) magnification of $5000\times$.

Table 2
Examination of reaction conditions^a CH₃CH₂CH₂CO₂H +
CH₃(CH₂)₂CH₂OH $\xrightleftharpoons[\text{H}_2\text{O}]{\text{cat.}}$ CH₃CH₂CH₂CO₂CH₂(CH₂)₂CH₃ + H₂O

Entry	Temperature (°C)	Catalyst (mol%)	H ₂ O (mmol)	Time (h)	Yield (%)
1	40	PS-SI, 10	0	72	89.6
2	80	PS-SI, 10	0	24	90.2
3	80	No cat.	5.56	48	3.2
4	80	PS-SI, 1	5.56	48	73.5
5	80	PS-SI, 5	5.56	48	90.7
6	80	PS-SI, 10	5.56	48	86.9
7	80	PS-SI, 10	27.8	48	41.3
8	80	PS-SI, 10	1.67	48	90.3

^a The reactions were carried out with *n*-butyric acid 1.26 mmol and *n*-butanol 1.26 mmol while the mixture was stirred. The products were analysed by GC by using *n*-decane as internal standard.

Table 3
Esterification of different reactants with 5 mol% PS-SI^a



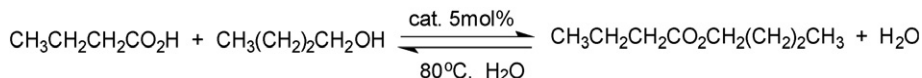
Entry	R	R'	Yield (%)
1	CH ₃ –	<i>n</i> -C ₄ H ₉ –	92.6
2	C ₃ H ₇ –	C ₂ H ₅ –	83.7
3	C ₃ H ₇ –	<i>n</i> -C ₄ H ₉ –	90.7
4	C ₃ H ₇ –	<i>n</i> -C ₃ H ₇ –	91.3
5	C ₃ H ₇ –	Cl-CH ₂ CH ₂ –	83.0
6	C ₃ H ₇ –	<i>i</i> -C ₃ H ₇ –	70.0
7	C ₃ H ₇ –	<i>s</i> -C ₄ H ₉ –	53.7
8	C ₃ H ₇ –	<i>t</i> -C ₄ H ₉ –	– ^b
9	<i>n</i> -C ₇ H ₁₅ –	<i>n</i> -C ₁₂ H ₂₅ –	44.5

^a The reactions were carried out with equimolar carboxylic acids and alcohols (1.26 mmol), PS-SI 5 mol% and H₂O (5.56 mmol) while the mixture was stirred at 80 °C for 48 h. The products were analysed by GC by using *n*-decane as internal standard.

^b Most of *t*-butyl alcohol was converted to 2-butylene.

loss between 250 and 370 °C may be attributed to the loss of the long chain alkyl groups –(CH₂)₁₀– and that above 400 °C the decomposition of the backbone of polystyrene. The results imply that PS-SI as the polymer acid catalyst will be stable below 160 °C.

Table 4
Comparison of different solid-state acids^a



Catalyst	Acidic loading (mmol/g)	Reaction time (h)	Yield (%)
Nafion NR50	0.89	24	86.9
		48	86.2
NKC-9	4.74	48	81.2
PPFSI	3.21	48	60.6
PS-SI	3.19	48	90.7

^aThe reactions were carried out with equimolar carboxylic acid and alcohol (1.26 mmol), catalyst 5 mol% and H₂O (5.56 mmol) while the mixture was stirred at 80 °C. The products were analysed by GC by using *n*-decane as internal standard.

3.6. Catalytic activity of PS-SI

To assess the catalytic activity of PS-SI, direct esterifications of equimolar carboxylic acids and alcohols in excess water catalyzed by PS-SI were investigated.

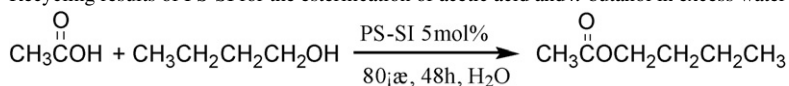
As shown in Table 2, 5 mol% PS-SI smoothly catalyzed the esterification of equimolar *n*-butyric acid and *n*-butanol in excess water (5.56 mmol) at 80 °C for 48 h and even in the presence of 1 mol% PS-SI excellent catalytic ability was observed (entry 4). The yield of ester was only 3.2% in without catalyst (entry 3). It was found that the amount of water was very important for obtaining good results. Increasing the amount of water was unfavorable for the esterification and large amount of water shifted the chemical equilibrium to the hydrolysis of ester (entry 7) even though 10 mol% amount of PS-SI was used. Suitable amount of water might be favorable for the formation of some kind of emulsion droplets from catalytic amount of the catalyst and reaction substrate [6]. On the other hand, when the esterification of equimolar *n*-butyric acid and *n*-butanol was carry out without any solvents (neat conditions, entry 2) the yield of ester reached 90.2% at 80 °C for 24 h.

The results of esterification of different structure acids and alcohols catalyzed by 5 mol% PS-SI in excess water are shown in Table 3. It can be seen that PS-SI was highly effective for the esterification of primary alcohols with carboxylic acids (entries 1–5) and its catalytic activity was not satisfactory for secondary alcohols (entries 6 and 7). In the case of a tertiary alcohol, it was found that the most of *t*-butyl alcohol was converted into 2-butylene (entry 8). On the other hand, PS-SI showed low catalytic activity for the long-chain acids (entry 9).

Table 4 lists the results of catalytic esterification of equimolar *n*-butyric acid and *n*-butanol over different solid acids. It can be seen from Table 4 that Nafion NR50 showed the highest catalytic activity of all the catalysts and for 24 h the yield of ester reached to 86.9%, which might be attributed to its high Hammett acidic strength. The ion-exchange resin catalyst NKC-9 also showed better catalytic activity for this esterification because of its higher acidic loading.

Under the same experiment conditions, the best result was obtained with PS-SI as the catalyst and it was a much better catalyst than PPFSI. PPFSI was a kind of polymer-supported sulfonimide catalyst without long-chain alkyl group [13]. This result indicates that the long chain alkyl group of the catalyst

Table 5
Recycling results of PS-SI for the esterification of acetic acid and *n*-butanol in excess water^a



Run	Yield (%)
1	92.6
2	92.2
3	92.4
4	90.6
5	93.0

^aThe reactions were carried out with equimolar carboxylic acid and alcohol (1.26 mmol), PS-SI 5 mol% and H₂O (5.56 mmol) while the mixture was stirred at 80 °C for 48 h. The products were analysed by GC by using *n*-decane as internal standard.

played an important role in the catalytic reaction. Some kind of emulsion droplets from the catalyst and reaction substrate might be formed under the experimental conditions and that would be favorable to shifting the chemical equilibrium to the formation of ester [6].

The reusability is an important feature of heterogeneous catalyst in green chemistry. PS-SI was easily separated from the reaction mixture by filtration and could be reused more than five times without any loss of catalytic activity (Table 5).

4. Conclusions

A new solid acid, PS-SI, has been prepared and characterized by FT-IR, solid state ¹⁹F NMR, SEM and TGA. The acid strength test showed that PS-SI was a strongly acidic polymer catalyst. Direct esterification of equimolar carboxylic acids and alcohols in excess water were selected as the model reaction to test its catalytic activity. The results indicated that PS-SI was a highly effective and reusable heterogeneous catalyst.

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