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Syntheses of high molecular weight aliphatic polyesters in 1-alkyl-3-methylimidazolium ionic liquids

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Abstract

High molecular weight aliphatic polyesters were synthesized, for the first time, in 1-alkyl-3-methylimidazolium ionic liquids via two-step polycondensation. An oligoester with diol/diacid ratio higher than unity was essential for achieving high molecular weight product. Moreover, the molecular weight of the resulting polyesters was found to depend on the activity of the catalyst in the ionic liquids and the miscibility of aliphatic polyester/ionic liquid. The former factor was dominated by the anion of the ionic liquids. The latter factor could readily be tuned by varying the anion and/or the cation of the ionic liquids. A clear correlation was found between the miscibility of aliphatic polyester/ionic liquid and the extent to which their solubility parameters matched.

Keywords: Aliphatic polyester; Ionic liquid; Polycondensation

1. Introduction

Polyesters are a family of polymers of increasing significance because of their biodegradability. The most common methods for synthesis of polyesters include bulk polycondensation under high temperature [1,2] and solution polycondensation in high-boiling organic solvents [3]. However, these methods require either a substantial amount of energy or hazardous volatile solvents. Ionic liquids are a category of thermally stable ionic compounds which have low volatility and are capable of dissolving many organic or inorganic substances [4,5]. These characteristics have made them as potential green solvents for preparing polymers with various polymerization mechanisms.

Free radical polymerizations in ionic liquids lead to higher overall rates and higher molecular weight of the products than those in organic solvents, because of the considerably higher k_p/k_t value of the reaction in ionic liquids [6,7]. ATRP could also benefit from using ionic liquids, as the catalysts are soluble in ionic liquids and are easy to be separated from the polymer [8,9]. Other controlled radical polymerizations, such as reverse atom transfer polymerization [10], nitroxide-mediated controlled polymerization [11] and reversible addition–fragmentation chain transfer polymerization [12] have also been successfully carried out in ionic liquids. The cationic polymerization of styrene in *N*-butyl-*N*-methylpyrrolidonium bis(trifluoromethanesulfonyl)amide has been found to proceed in a living way [13]. Furthermore, polycondensations in ionic liquids have been exploited to synthesize high molecular weight polyamides and polyimides [14,15] and other high performance polymers [16] under milder reaction conditions.

However, the synthesis of polyesters in ionic liquids has been confronted with some challenges. The enzymatic ring-opening polymerization of ε-caprolactone and ester-exchange polymerization between the derivatives of various diacids and 1,4-butanediol were investigated in imidazolium-based ionic liquids by Heise and other authors [17–20]. The molecular weights of poly(ε-caprolactone) did not exceed 10,000 g/mol. And the highest molecular weight of the polyesters synthesized from ester-exchange polymerization only reached

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5400 g/mol. The authors suggested that low molecular weight were caused by the immiscibility of the polyesters with the ionic liquids when their molecular weights reached a certain limit. Fradet and coworkers reported the synthesis of poly(glycolic acid) (PGA) in imidazolium-based ionic liquids either directly from glycolic acid or from preformed oligomer [21]. Low PGA yields were obtained by direct method because of the severe evaporation of the monomer at high reaction temperature. The PGA with the highest degree of polymerization up to 45 was obtained by the postpolymerization of the preformed oligomer in 1-butyl-3-methylimidazolium bis(trifluoromethanesulfonyl)amide. The author claimed that the precipitation of PGA in reaction medium and the low efficiency of metal catalysts in the ionic liquids limited the achievable molecular weight. Therefore, the miscibility of polyester/ionic liquid and the activity of catalysts in ionic liquids are the two factors which deserve attention when we try to synthesize high molecular weight polyesters in ionic liquids.

This work intends to define the two parameters mentioned above through a series of experiments in order to fully explore this promising green chemistry route for synthesis of polyesters. Our results indicate that the effectiveness of Sn^{2+} catalyst in ionic liquids can readily be tuned by varying the anion and/or cation of ionic liquid, and that the miscibility of aliphatic polyester/ionic liquid can be optimized by matching the Hildebrand solubility parameters [22] of the ionic liquid and the resulting polyester. These findings enable us to synthesize high molecular weight aliphatic polyesters in ionic liquids at high yield.

2. Experimental details

2.1. Materials

All of the ionic liquids used in this work were synthesized by literature procedures [23–25]. The purity of the ionic liquids was confirmed by ^1H NMR and elemental analysis. The content of halide anions in the ionic liquids proved below the detection limit of 0.1 M silver nitrate solution. All of the ionic liquids were dried under vacuum before usage to eliminate the water absorbed from air. Sebacic acid, adipic acid, succinic acid and hexanediol were purified by recrystallizing from water. Ethylene glycol was purified by fractional distillation under reduced pressure. $SnCl_2 \cdot 2H_2O$ was purchased from Beijing Dong Fang Long Shun Chemical Synthetic Technology Center Corp. and used as-received.

2.2. Syntheses of oligoesters

Oligo(hexamethylene sebacate) (oligoester 10,6) was synthesized by bulk polycondensation. In a typical procedure, 12.54 g (0.062 mol) sebacic acid and 7.77 g (0.066 mol) hexanediol were introduced into a 100 mL three-necked round bottom flask equipped with mechanical stirrer, gas inlet and gas outlet. A gentle nitrogen stream was bubbled into the mixture. The flask was then heated in an oil bath to about 160 °C over a period of 10–11 h. The reaction mixture, after cooling, was smashed into bits, which were used in subsequent polycondensations without further treatment. The weight-average molecular weight (M_w) of the obtained oligoester was determined to be lower than 5000 by gel permeation chromatography (GPC).

Oligo(hexamethylene adipate) (oligoester 6,6) was synthesized by identical procedure as described above. The M_w of oligoester 6,6 was 5400. Oligo(ethylene succinate) (oligoester 4,2) was synthesized with similar procedure, but at higher temperature (170–185 °C) over 7 h. The M_w of oligoester 4,2 was 8000.

2.3. Postpolycondensations in ionic liquids

For a typical postpolycondensation of oligoesters in ionic liquids, 2.15 g of oligomer, 4.00 g ionic liquid and 0.01 g $SnCl_2 \cdot 2H_2O$ were added to a 10 mL vial. The apparatus with reactants was evacuated and filled with dry nitrogen for several times. The polymerization was conducted under vacuum (~50 Pa) at 160 °C. Customized mechanical stirring equipment was used. After 24 h, the reaction mixture was held unstirred at the reaction temperature for 10–15 min until phase separation completed, if phase separation occurs. Then the vial was quenched in an ice water to freeze the composition of each phase. The upper phase and the lower phase were collected and analyzed, respectively.

2.4. Bulk syntheses of polyesters

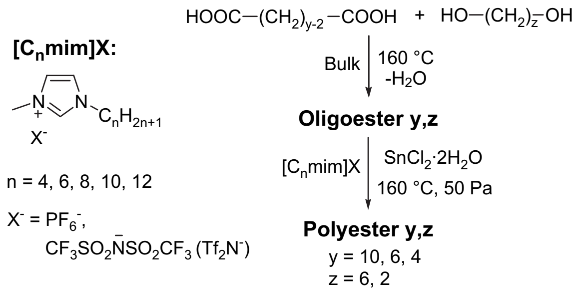
Poly(hexamethylene sebacate) (polyester 10,6), poly(hexamethylene adipate) (polyester 6,6) and poly(ethylene succinate) (polyester 4,2) were synthesized from their oligomers by bulk polycondensation at 160 °C under vacuum for 6–8 h with 0.5 wt% $SnCl_2 \cdot 2H_2O$ as catalyst. The M_w of the resulting polyesters was in a range of 4.0×10^4–6.0×10^4.

2.5. Miscibility tests

Ionic liquid (40 wt%) was added into the vial and further dried at 120 °C under vacuum (<20 Pa) for 15 min, then cooled to room temperature, followed by an addition of 60 wt% polyester. Totally weighed 0.4–1.2 g, the mixture was stirred under nitrogen atmosphere at increasing temperature up to 160 °C. The mixing process was monitored by visual inspection.

2.6. Characterization methods

The molecular weight analyses were performed using a Polymer Laboratories gel permeation chromatograph (PL GPC50) equipped with a RI detector. The solvent used was tetrahydrofuran or chloroform with a flow rate of 1 mL min^{-1}. The columns used are PLgel mixed-B for tetrahydrofuran eluent and PLgel mixed-C for chloroform eluent, all calibrated using polystyrene standards. Peaks of polyesters in the GPC chromatograms were integrated excluding those of the cyclic



Scheme 1. Procedure for two-step polycondensation.

oligomers. ^{1}H NMR spectra were recorded in $CDCl_3$ solution on a Bruker Advance DRX-500 NMR spectrometer. Inductively coupled plasma (ICP) data were collected on a JY Ultima ICP spectrometer. Elemental analysis was performed with Elementar Vario El.

3. Results and discussion

Polyesters (Scheme 1) were synthesized in ionic liquids by the postpolycondensation of oligoesters under vacuum (~50 Pa) at 160 °C using a simple catalyst, $SnCl_2 \cdot 2H_2O$. Oligomers, instead of diacid and diol, were used as the starting materials to eliminate the large amount of water generated in the initial stage of the formation of the oligomers. A large amount of water in the system may affect the performance of Lewis acid catalysts and the stability of fluorinated inorganic anions, such as PF_6^- anion, especially at elevated temperatures [4,5].

3.1. The diol/diacid ratio of the oligomers

The diol/diacid ratio of the preformed oligomers is an important factor affecting the molecular weight of the resulting polyesters. When the diol/diacid ratio was lower than unity, only low molecular weight product was obtained no matter which ionic liquid was used. This phenomenon may be attributed to the fact that excessive high-boiling sebacic acid could hardly be evacuated during the reaction, which restrains the polycondensation equilibrium from moving towards the higher molecular weight polyester. Therefore, all the oligoesters used in this work had a diol/diacid ratio higher than 1.01 (the diol/diacid ratio was calculated from ^{1}H NMR spectra).

3.2. The activity of Sn^{2+} catalyst in the ionic liquids

The activity of Sn^{2+} catalyst is dominated by the anion of ionic liquids as reflected in the molecular weight (Table 1) of the polyesters. In PF_6^- ionic liquids, only relative low molecular weight products ($M_w < 1.8 \times 10^4$) were obtained, which was similar to that obtained in bulk polymerization with no catalyst ($M_w \sim 1.7 \times 10^4$). In Tf_2N^- ionic liquids, the molecular weight increased significantly ($M_w > 2.8 \times 10^4$). Although the results were for a different class of polyester, Fradet and coworkers reported that the average degree of polymerization of PGA synthesized in $[C_4mim]PF_6$ was lower than that in $[C_4mim]Tf_2N$ [21]. They also claimed that the low catalytic activity of metal cation in ionic liquid was caused by the preferential combination of the metal cation with the anion of the ionic liquid. However, we suppose herein a somewhat different mechanism according to Clark and Jones' work on hard and soft acid–base principle (HSAB) [26]. By kinetic studying, Clark and Jones found that the hard acidic metal ions accelerate the hydrolysis of the PF_6^- anion, and themselves are transferred into fluoride complexes $[MF_n]^{y-n}$. The higher the coordination number is, the less catalytically active the complex will be, as the hard acid–hard base interaction between the metal ion and the fluoride ion is very strong and prevent other ligands from coordinating to

Table 1
Postpolycondensations of oligo(hexamethylene sebacate) in ionic liquids

Entry[a]	Ionic liquid	M_w[b] (10^4)	PDI	Yield[c] (%)	P. S.[d]	RU/ILs[e]		Sn[f] (ppm)
						UP	LP	UP/LP
1	$[C_4mim]PF_6$	1.8	2.10	ND	I	94.1/5.9	0.7/99.3	82/1325
2	$[C_6mim]PF_6$	1.5	1.96	ND	I	85.1/14.9	1.3/98.7	153/1323
3	$[C_8mim]PF_6$	1.4	1.85	ND	I	80.9/19.1	4.8/95.2	114/1308
4	$[C_{10}mim]PF_6$	0.9	1.75	ND	II	–	–	–
5	$[C_{12}mim]PF_6$	0.8	1.71	83.4	III	–	–	–
6	$[C_4mim]Tf_2N$	2.8	1.97	ND	I	77.6/22.4	2.5/97.5	1008/308
7	$[C_6mim]Tf_2N$	3.8	1.67	90.0	III	–	–	–
8	$[C_8mim]Tf_2N$	7.1	1.65	86.2	III	–	–	–
9	$[C_{10}mim]Tf_2N$	8.7	1.75	92.8	III	–	–	–
10	$[C_{12}mim]Tf_2N$	7.7	1.78	96.3	III	–	–	–

[a] General polymerization conditions: 35 wt% oligo 10,6, 65 wt% ionic liquid and 0.5 wt% $SnCl_2 \cdot 2H_2O$ reacting at 160 °C under 50 Pa for 24 h.

[b] Weight-average molecular weight (M_w) and polydispersity index (PDI) measured by GPC calibrated with polystyrene standards. Note that no purification was performed to the samples.

[c] Yield was calculated as the ratio between the purified product's weight and the initial oligoester's weight. ND = not determined.

[d] P.S. = phase separation at reaction temperature, I = completely phase separation, II = uncompleted phase separation even wait for 30 min, III = no phase separation.

[e] RU = Repeat unit, UP = Upper phase (polyester phase), LP = Lower phase (ionic liquid phase), the molar ratios of RU to ILs were calculated from ^{1}H NMR.

[f] Residual Sn contents measured by ICP.

the metal ion. The coordination number n is determined by the ratio of metal catalyst/PF_6^- anion. In our case, the PF_6^- anion was overwhelming excessive to the Sn^{2+}, which is a typical hard acid. So we suppose that the Sn^{2+} cation was changed into totally fluorinated complex in the PF_6^- ionic liquids, which was inactive for catalysis. In contrast, the covalent C–F bond of Tf_2N^- anion was very strong and therefore remained intact during the reaction. As a result, Sn^{2+} cation was still catalytically active in Tf_2N^- ionic liquids. However, more detailed researches are needed to prove the above suppositions. Alike polyester 10,6, high molecular weight ($M_w > 5.0 \times 10^4$) polyester 6,6, and 4,2 were synthesized from their oligomers in Tf_2N^- ionic liquids (not shown here).

3.3. The miscibility between ionic liquids and polyesters

The miscibility between the ionic liquid and the resulting polyester, as expected, also plays a large role in controlling the molecular weight of the polymer. (Table 1) For instance, in PF_6^- series, better miscibility brought the molecular weight of the polyesters down. Conversely, in Tf_2N^- series, the trend was reversed. The ICP measurements (Table 1) of residual Sn in the upper and lower phases were performed when phase separation occurred. In the cases of PF_6^- ionic liquids substituted with short chain alkyl group (Table 1, entries 1–3), most of the Sn catalyst was found in the ionic liquid phase, and was expected to be transferred into inactive species as discussed above. Therefore there was only a small fraction of the catalysts in the polyester phase and remained active in catalyzing the reaction. However, homogeneous or nearly homogeneous reaction in PF_6^- ionic liquids substituted with long chain alkyl group (Table 1, entries 4, 5) would allow all of the Sn catalysts to form inactive species. As a result, the molecular weight of the polymers was particularly low. In Tf_2N^- series, whose anion did not suppress the activity of the catalysts, more catalyst was available to the end group of the polyester in the homogeneous system than in the heterogeneous one. Therefore, the molecular weight of the polyesters tended to increase with increasing miscibility between ionic liquid and polyester.

3.4. Structure–miscibility relationship

The fact that the miscibility varied regularly according to the structure of the ionic liquids made us go further. A series of miscibility tests were performed and extended to include more polyester (Table 1). The miscibility was improved by increasing the length of the alkyl chain on the cation of the ionic liquid. Furthermore, the miscibility was remarkably improved by replacing the ionic liquid anion from PF_6^- to Tf_2N^- anion, as shown by the experimental observation and ^1H NMR calculations (Table 1). Polyesters with less methylene units in their structural units showed better miscibility with ionic liquids than their homologues with more methylene units did. For instance, polyesters 6,6 and 4,2 were miscible with $[C_4mim]Tf_2N$ and/or $[C_{10}mim]PF_6$ at 160 °C, with which polyester 10,6 was immiscible at the same temperature.

Interestingly, a clear correlation was found between the miscibility and the difference in Hildebrand solubility parameters (δ_H) of the ionic liquids and the polyesters. δ_H has been widely used for prediction and understanding of the miscibility between polymers and small molecular solvents as well as between different polymers [27]. Two components become miscible when the difference between their δ_Hs is adequately small. The curves derived from Lee and Lee experimental data (Fig. 1a) [28] indicate that PF_6^- ionic liquids have much higher δ_H than their Tf_2N^- counterparts do. In addition, δ_Hs of both series decrease gradually as the alkyl substituent length increases [28]. The δ_H values of the aliphatic polyesters in amorphous phase were calculated by a group contribution technique (Eq. (1)) [22].

$$\delta_H = \rho \sum F_i / M \tag{1}$$

where ρ is polymer density, F_i is molar attraction constant of each group at 298.15 K, and M is molar mass of the repeat unit. The values of F_i used for calculation were referenced to a handbook [22]. Here $F_{CH_2} = 269.0\ J^{1/2}\ cm^{-3/2}$, $F_{COO} = 668.2\ J^{1/2}\ cm^{-3/2}$. And the density data were calculated according to literature equation (Eq. (2)) [29].

$$\rho = B(T)(N_{COO}/M) + I(T) \tag{2}$$

where $B(T)$ and $I(T)$ are both functions of the temperature, and equals 28.2168 and 0.8439, respectively, at 298.15 K. N_{COO} is the number of ester linkage in the repeating unit of the polymer, and equals 2 herein. As shown in Fig. 1b, the δ_H of aliphatic polyesters decreases with the carbon number in their repeating unit. The difference in Hildebrand solubility parameter ($\Delta\delta_H$) is illustrated in Fig. 1c for different combinations of ionic liquid and polyesters. As expected, polyester/ionic liquid system changes from miscible (Fig. 1d) to immiscible (Fig. 1e) if its $\Delta\delta_H$ exceeds a certain limit (Fig. 1c). The limit of $\Delta\delta_H$ lies in the range of 7.9–8.0 MPa$^{1/2}$ for polyester 10,6, and 8.6–9.4 MPa$^{1/2}$ for polyester 6,6, and beyond 10.7 MPa$^{1/2}$ for polyester 4,2. The reason for the existence of different limits for polyesters with different structures is not known. It is satisfactory that this single-parameter approach offers us a basic parameter to design a miscible system for ionic liquids and polyesters, though it has problems in revealing the details of the interactions between ionic liquid and polyester. A more detailed picture of individual interactions is to be expected if multiple-parameter approaches are used, such as Hansen parameters [22] or Kamlet–Taft parameters from solvatochromic methods [30] or interaction parameters from inverse GC measurement [31].

3.5. Purification of the polyesters

Polyesters in the homogeneous reaction systems or the upper phases of heterogeneous reaction systems are only soluble above their melting points. When the temperature dropped below their melting points, the polyesters would be separated from the ionic liquids because of the crystallization. This

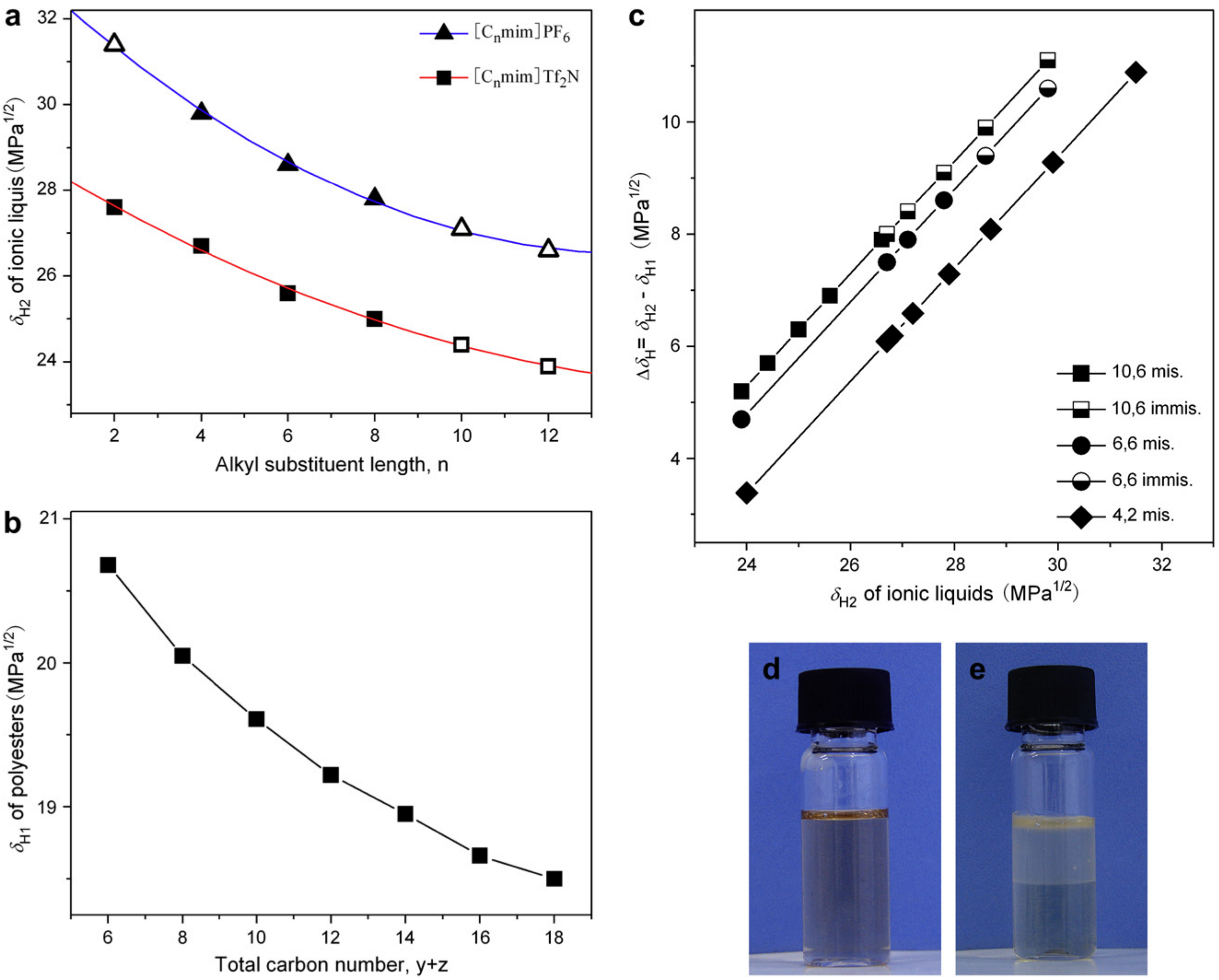


Fig. 1. (a) Solubility parameters δ_{H_2} of ionic liquids (▲ and ■ are literature values, △ and □ are estimated values, for estimation details see Supplementary information). (b) Calculated solubility parameters δ_{H_1} of aliphatic polyester *y*,*z*. (c) Difference between δ_{H_2} and δ_{H_1} against δ_{H_2}, enclosed symbols mean that the system is miscible (d) and half enclosed symbols mean immiscible (e) at 160 °C.

crystallization-induced phase separation simplified the purification process. By simply removing the ionic liquid using methanol, then washing for 4 times, high-purity (>99%) polyester 10,6 (Fig. 2) was obtained at a yield up to 96.3%. The interesting fact was that the purification by methanol eliminated the cyclic oligomers but nearly no linear oligomers (Fig. 3). The molecular weight distributions of the polyester before and after purification were almost the same. This

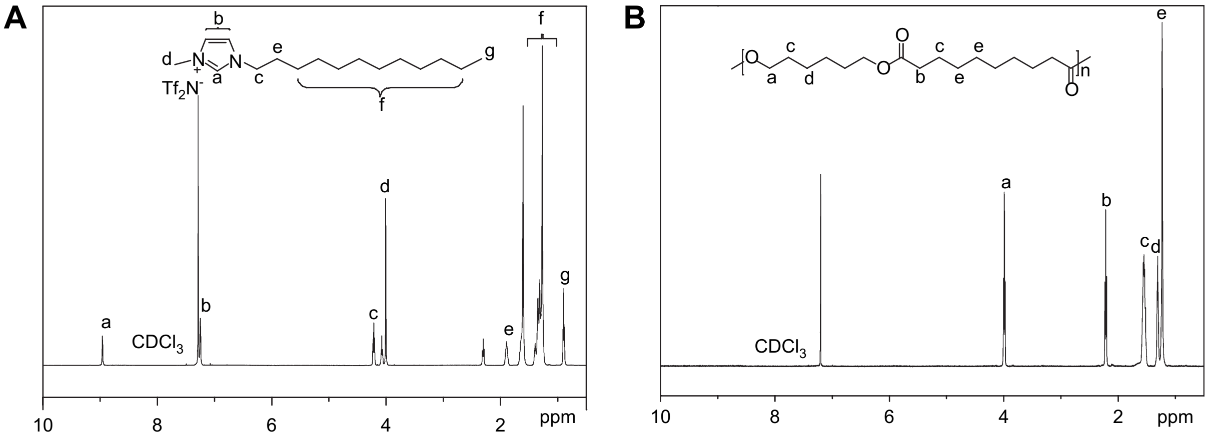


Fig. 2. ^{1}H NMR spectra of unpurified reaction mixture (A) and purified polyester 10,6 (B).

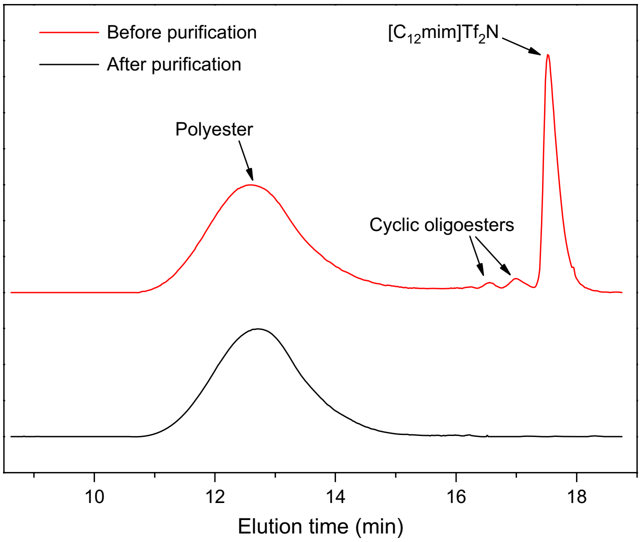


Fig. 3. The GPC chromatograms of the polyester 10,6/$[C_{12}mim]Tf_2N$ before and after purification.

observation also demonstrated that the ionic liquids lost their ability to dissolve the polyesters when the mixture was cooled to below the melting points of the polyesters.

4. Conclusions

Experimental results illustrate that high molecular weight aliphatic polyesters of value in commercial application could be synthesized by this green chemistry route in ionic liquids. Keeping the diol/diacid ratio of the oligoesters higher than unity was essential to the growth of the molecular weight. The activity of catalyst in ionic liquids and the miscibility of aliphatic polyester/ionic liquid were proved to be two key factors controlling the molecular weight of the polyesters. The Sn^{2+} catalyst remained active in Tf_2N^- ionic liquids, and was substantially suppressed in PF_6^- ones. The miscibility was found to be a function of both the anion and the cation of the ionic liquids. Hildebrand solubility parameter was identified as a semi-quantitative criterion in choosing ionic liquids for the polyesters with a given chemical composition. Using multiple-parameter approach may provide more details of individual interactions between the ionic liquid and the polyester. And this work is still underway in our laboratory. Polyester with high purity was obtained at high yield by simple precipitating and washing process.

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Appendix. Supplementary information

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.polymer.2007.11.044.

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