

Kinetic Study of the Atom Transfer Radical Polymerization of *n*-Docosyl Acrylate

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ABSTRACT: The atom transfer radical polymerization (ATRP) of *n*-docosyl acrylate (DA) was studied at 80°C in *N,N*-dimethylformamide using the carbon tetrabromide/FeCl₃/2,2'-bipyridine (bpy) initiator system in the presence of 2,2'-azobisisobutyronitrile (AIBN) as the source of reducing agent. The rate of polymerization exhibits first-order kinetics with respect to the monomer. The linear relationship between the molecular weight of the resulting poly(*n*-docosyl acrylate) with conversion and the narrow polydispersity of the polymers indicates the living characteristics of the

polymerization reaction. The significant effect of AIBN on the ATRP of DA was studied keeping [FeCl₃]/[bpy] constant. A probable reaction mechanism for the polymerization system is postulated to explain the observed results. © 2005 Wiley Periodicals, Inc. *J Appl Polym Sci* 97: 2147–2154, 2005

Key words: atom transfer radical polymerization; *n*-docosyl acrylate; transition metal catalyst; polydispersity; comb-like polymers

INTRODUCTION

Free radical polymerization is one of the most important commercial processes for the synthesis of high-molecular-weight polymers, because a wide variety of monomers can be polymerized under relatively simple experimental conditions.¹ The importance of radical polymerization in industry is mainly due to its high tolerance of impurities, easy implementation, and good reproducibility. But conventional free radical polymerization has a significant drawback, which is related to the reactivity of the propagating free radical and its tendency to undergo different termination reactions. The resultant materials are therefore polydispersed with very limited control over molecular weight (MW) and architecture. Important aspects of polymer chemistry have been the precise control of polymerization reactions that leads to well-defined polymers in terms of MW and molecular weight distribution (MWD), structure of end groups, stereoregularity, monomer sequence, and topology. Control of these parameters is extremely important for the synthesis of new speciality polymers, because the physical and chemical properties of these polymers are structure dependent.^{2,3}

Atom transfer radical polymerization (ATRP) is a new and efficient method for synthesis of polymers with well-defined MW, low polydispersity, and desired architecture.^{4,5} Although there are several approaches to control free radical polymerization, the best method is required to achieve good control over MWs, MWD, and polymer architecture.^{6–10} An advantage of transition metal catalyzed ATRP is that it does not require strict experimental conditions. Therefore, this living process provides a new approach for synthesis of polymers with controlled MW and MWD.^{6,10–12} Atom transfer radical polymerization has been applied to a wide range of monomers, including many different functional monomers, for the synthesis of a diverse array of polymers with varied composition and architecture. Currently, attention has been shifted toward iron-catalyzed ATRP because of its lower cost and similar activity to their ruthenium analogs.^{13–16} Halide ions have been used as complexing ligands in iron catalyzed ATRP in both normal and reverse ATRP.^{14,17}

Considering the toxicity of the halide species and the easy oxidation of the catalyst by oxygen in air, Matyjaszewski and coworkers^{18,19} and Teyssie et al.²⁰ reported the reverse or alternative ATRP systems, respectively. In reverse ATRP systems the catalyst is used in its higher oxidized state ($M_n^t + 1X$) and the initiator is a classical radical initiator such as 2,2'-azobisisobutyronitrile (AIBN).^{13,21,22} The initiation step is the decomposition of the radical initiator. The radical can then react with the monomer to create a growing chain or directly with the catalyst to form the

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reduced species M_n^+ and dormant species.²² The reverse ATRP initiated by peroxide sometimes behaves differently than that based on the azo initiators. In reverse ATRP, the initiation mechanism with benzoyl peroxide (BP) is different from that of AIBN because there is a redox reaction between BP and the formation of reduced transition metal species.²²

Comb-like polymers with pendent alkyl side chains of specific length have been extensively used as flow improvers/pour point depressants and wax dispersants for the transportation of waxy crude oils.^{23,24} The high conversion free radical polymerization of *n*-alkyl (meth)acrylates shows a strong gel effect.²⁵ Therefore, in high-conversion polymerization, considerable deviation from the normal kinetic parameters with a broadening of MWD is observed. Although acrylates can be polymerized anionically, the preparation of well-defined polymers is hindered by a number of side reactions.²⁶ Thus, the idea of combining the advantage of both living polymerization and radical polymerization has attracted much attention. Matyjaszewski et al.^{27,28} reported that the heterogeneity of the reaction mixture in the ATRP of *n*-dodecyl acrylate and *n*-octadecyl methacrylate (OMA) is due to the poor solubility of the catalyst complex formed with linear tridentate amine and metal complex in both the monomers and the nonpolar solvents. This resulted in ill control of the polymer molecular weights with high M_w/M_n .

To synthesize polymers of long chain alkyl acrylates and methacrylates with controlled architecture and narrow polydispersity, recently we reported the ATRP of OMA.²⁹ This article describes the synthesis of well-defined poly(*n*-docosyl acrylate) (PDA) with narrow polydispersity using a novel initiating system that comprises a catalytic amount of AIBN as radical source, carbon tetrabromide (CBr_4) as the initiator, and $\text{FeCl}_3/2,2'$ -bipyridine as the catalyst.

EXPERIMENTAL

n-Docosyl acrylate (DA) was prepared and purified as before.²⁵ The solvents and the initiator (AIBN) were purified by standard methods. The 2,2'-bipyridine (bpy), FeCl_3 , and CBr_4 were used without further purification.

The polymerization of DA was carried out in a dry glass ampoule with nitrogen (99.99% pure). The solution was deaerated by three freeze-pump-thaw cycles and sealed with a rubber septum. The tubes were placed in a thermostat at desired temperatures for a definite period of time. After the desired time period, precipitation with acetone containing traces of hydroquinone isolated the polymers. The polymers were then filtered, reprecipitated several times, and dried *in vacuo*. The percentage conversion was determined gravimetrically. MW and MWD of the polymers were

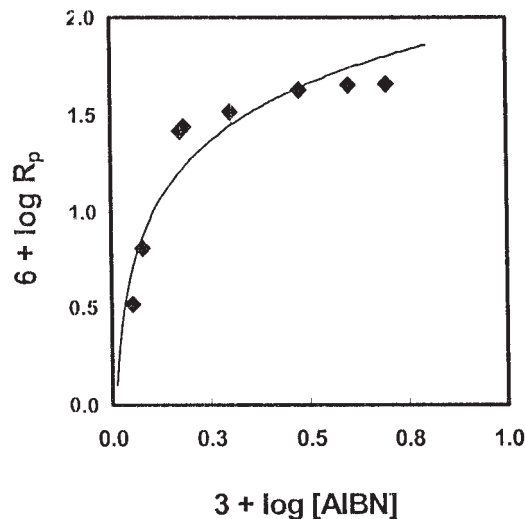


Figure 1 Relationship between $\log R_p$ and $\log [\text{AIBN}]$ for the ATRP of DA at 80°C in DMF. Conditions: $[\text{DA}] = 1.00 \text{ mol L}^{-1}$; $[\text{CBr}_4] = 5.00 \times 10^{-2} \text{ mol L}^{-1}$; $[\text{Fe(III)}] = 1.33 \times 10^{-3} \text{ mol L}^{-1}$; $[\text{bpy}] = 4.00 \times 10^{-3} \text{ mol L}^{-1}$.

determined using a Waters 515 gel permeation chromatograph equipped with three Styragel columns (HR1, HR3, and HR4) in series with a 2410 differential refractometer as the detector. The analysis was performed at room temperature with purified high-performance liquid chromatography grade tetrahydrofuran as eluent at 1 mL min^{-1} . Calibration was based on narrow molecular weight distribution polystyrene standards with the Waters Millennium 2.0 software package. Differential scanning calorimetry (DSC) was performed on a TA series DSC 2010 instrument at a heating rate of $10^\circ\text{C min}^{-1}$. The X-ray diffractogram of polymers was recorded on a Model JDX-11P3A JEOL diffractometer with a solid sample using a Ni filter with $\text{Cu-K}\alpha$ radiation at 35 kV and 10 mA in the wide angle range $2^\circ < \theta < 60^\circ$. The $^1\text{H-NMR}$ spectrum was recorded on a Bruker 300-MHz spectrometer in CDCl_3 with tetramethylsilane as an internal reference.

RESULTS AND DISCUSSION

The results of the polymerization of DA in *N,N*-dimethylformamide (DMF) under various experimental conditions were studied. Among the different components of polymerization, the reducing agent AIBN has a pronounced effect on the rate of polymerization (R_p). Under the experimental conditions used, no polymerization was observed below an AIBN concentration of $1.00 \times 10^{-3} \text{ mol L}^{-1}$. The effect of concentration of AIBN on the ATRP of DA was critically studied over a range from 1.00×10^{-3} to $1.50 \times 10^{-3} \text{ mol L}^{-1}$ (Fig. 1). R_p increases sharply with the increase of $[\text{AIBN}]$ up to $1.45 \times 10^{-3} \text{ mol L}^{-1}$ with a slope of 0.50 and beyond that the rate decreases. R_p was independent of $[\text{AIBN}]$

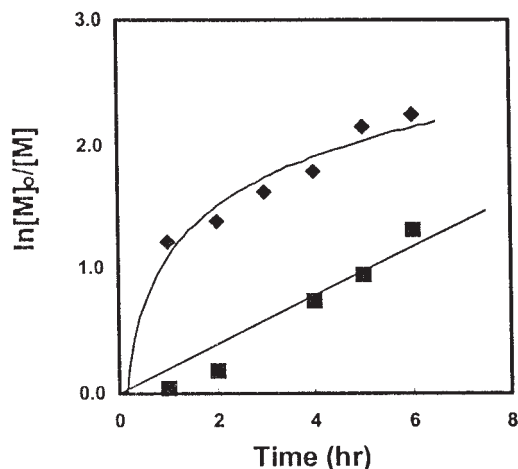


Figure 2 Effect of AIBN concentration on first-order kinetic plot for the ATRP of DA at 80°C in DMF. Conditions: [DA] = 0.50 mol L⁻¹; [CBr₄] = 0.10 mol L⁻¹; [Fe(III)] = 1.00 × 10⁻³ mol L⁻¹; [bpy] = 3.00 × 10⁻³ mol L⁻¹; [AIBN] = 5.00 × 10⁻³ mol L⁻¹ (■) and [AIBN] = 1.00 × 10⁻² mol L⁻¹ (◆).

of 2.00 × 10⁻² mol L⁻¹ and above. This indicates that the concentration of AIBN is critical for ATRP while keeping [Fe(III)]/[bpy] constant at 1:3. Therefore, the presence of a catalytic amount of growing radicals and dormant species is mandatory for the ATRP of DA. As anticipated, [AIBN] has a significant effect on the kinetics of polymerization of DA (Fig. 2). The highest [AIBN] led to the highest polymerization rate, corresponding to the highest initial radicals concentration and fastest reduction of Fe(III).³⁰ At low [AIBN], the linearity of the semilogarithmic plots of ln([M]₀/[M]) versus time indicated that the polymerization was first-order with respect to the monomer concentration.

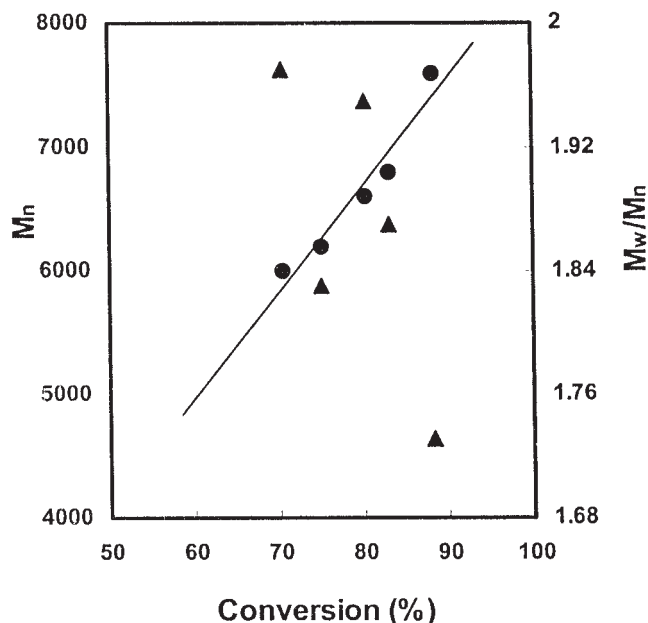


Figure 3 PDA molecular weight, M_n (●) and polydispersity index, M_w/M_n (▲) as a function of conversion in the ATRP of DA 80°C in DMF in the presence of AIBN as the source of reducing agent. Conditions: [DA] = 0.50 mol L⁻¹; [AIBN] = 1.00 × 10⁻² mol L⁻¹; [CBr₄] = 0.10 mol L⁻¹; [Fe(III)] = 1.00 × 10⁻³ mol L⁻¹; [bpy] = 3.00 × 10⁻³ mol L⁻¹.

The apparent rate constant (k_{app}) was 1.63 × 10⁻⁴ s⁻¹. This shows that the living radical concentration is constant throughout polymerization. With higher [AIBN], R_p increased rapidly and after a certain period it became steady.

To gain a better understanding of the ATRP mechanism, the effect of various parameters on the MW

TABLE I
Effects of the Concentration of DA, CBr₄, Fe(III), and AIBN on the Rate of Polymerization, Molecular Weight, Polydispersity Index, and Thermal Behavior of PDA Prepared by ATRP in a Nitrogen Atmosphere at 80°C in DMF

[DA] (mol L ⁻¹)	10 ² × [CBr ₄] (mol L ⁻¹)	10 ³ × [Fe(III)] (mol L ⁻¹)	10 ² × [AIBN] (mol L ⁻¹)	10 ⁵ × R_p (mol L ⁻¹ s ⁻¹)	M_n	M_w/M_n	m_p (°C)	ΔH_m (kJ mol ⁻¹)	ΔS_m (J K ⁻¹ mol ⁻¹)
1.00	10.00	1.33	2.00	4.53	6800	1.79	65.6	61.74	182.33
0.75	10.00	1.33	2.00	3.31	5600	1.86	61.0	50.42	150.96
0.50	10.00	1.33	2.00	2.16	4500	1.77	63.0	45.97	136.82
0.30	10.00	1.33	2.00	1.22	4000	1.54	64.4	43.65	129.39
1.00	8.00	1.00	2.00	2.83	3200	1.04	64.9	57.80	171.07
1.00	6.00	1.00	2.00	2.95	3500	1.16	61.5	49.98	149.42
1.00	4.00	1.00	2.00	3.39	3800	1.17	61.6	44.48	141.95
1.00	2.00	1.00	2.00	3.57	4200	1.19	61.8	45.97	137.31
0.50	10.00	5.00	0.50	1.14	8600	1.97	65.8	66.73	196.98
0.50	10.00	2.00	0.50	1.46	11300	1.64	67.1	67.98	199.90
0.50	10.00	1.00	0.50	1.69	14400	1.42	60.9	50.12	150.12
0.50	10.00	0.80	0.50	1.75	17100	1.13	62.2	49.40	147.46
0.50	10.00	0.20	0.50	1.94	7500	1.85	61.3	49.24	147.30
0.50	10.00	5.00	2.00	4.56	9000	1.65	63.9	59.60	176.91
0.50	10.00	0.67	2.00	4.42	6800	1.86	63.2	53.25	158.39
0.50	10.00	0.27	2.00	4.38	7200	1.79	64.3	63.39	187.94

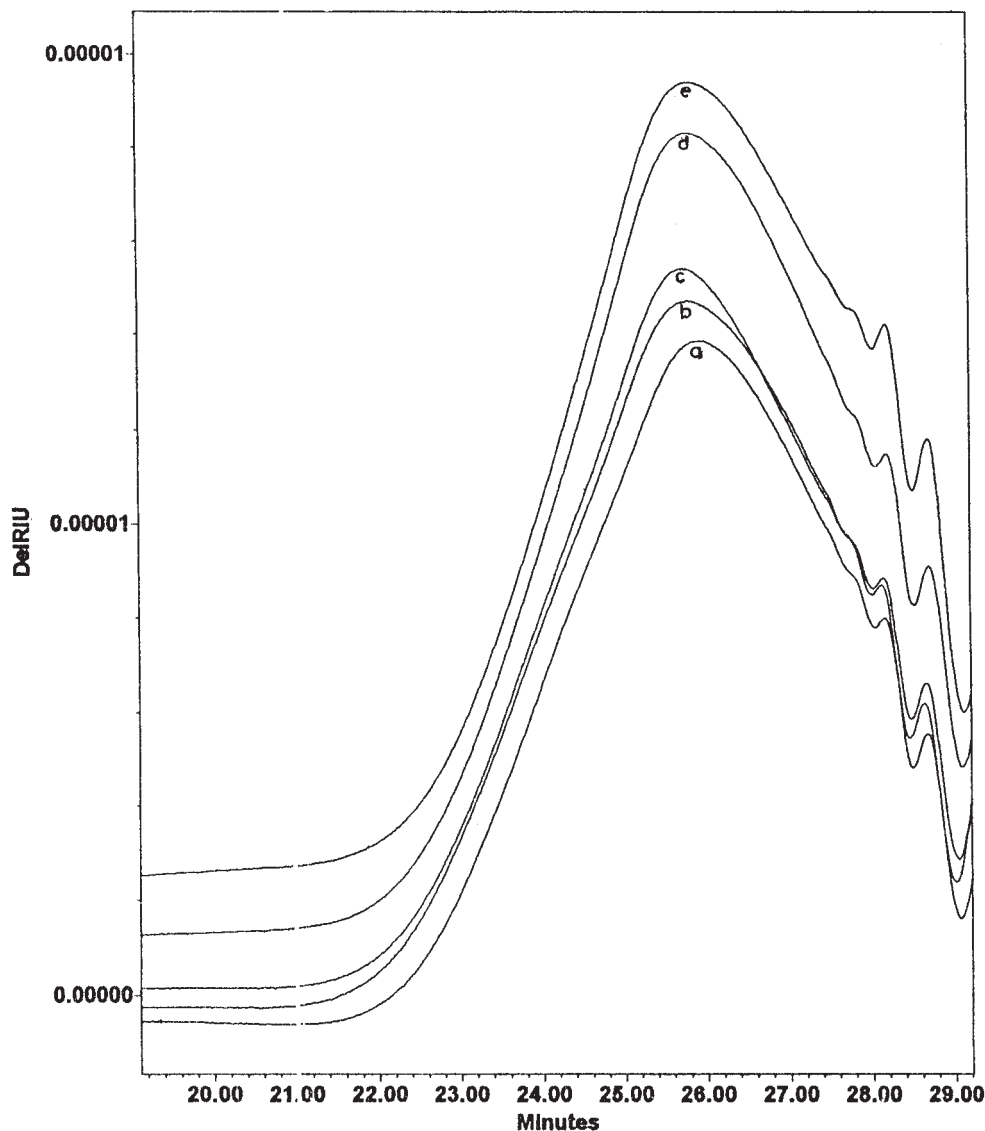
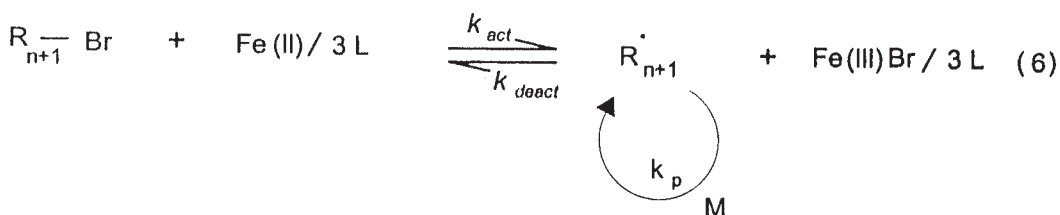


Figure 4 GPC traces (refractive-index detector) of PDA synthesized by ATRP of DA at 80°C in DMF in the presence of AIBN as the source of reducing agent. Conditions: [DA] = 0.50 mol L⁻¹; [CBr₄] = 0.10 mol L⁻¹; [AIBN] = 1.00 × 10⁻² mol L⁻¹; [Fe(III)] = 1.00 × 10⁻³ mol L⁻¹; [bpy] = 3.00 × 10⁻³ mol L⁻¹. Conversions for curves: (a) = 70.4%; (b) = 74.90%; (c) = 80.30%; (d) = 83.15%; (e) = 88.30%.

and M_w/M_n was studied. GPC studies have established the effect of variation of [DA], [CBr₄], [Fe(III)], and [AIBN] on $M_{n(\text{GPC})}$ and M_w/M_n of PDA and the values are presented in Table I. The number average molecular weight ($M_{n(\text{GPC})}$) of PDA increases with the increase of [DA] and decreases with the increase of [CBr₄]. The higher value of M_w/M_n with increasing [DA] may be due to branching reactions or to the gel effect.²⁰ The branching may be due to the extraction of a large number of hydrogen atoms from the long alkyl group of the alkyl chain. Molecular weights remained unchanged up to [AIBN] of 5.00 × 10⁻³ mol L⁻¹ and above this concentration AIBNs participate in the determination of the $M_{n(\text{GPC})}$ and MWD by creating the initiating radicals and in the determination of the cat-

alytic activity by changing the ratio Fe(III)/Fe(II). There was no significant effect of [Fe(III)] on $M_{n(\text{GPC})}$ and M_w/M_n of PDA. $M_{n(\text{GPC})}$ values are nearly same while M_w/M_n remains quite narrow at different [Fe(III)]. Figure 3 shows that the $M_{n(\text{GPC})}$ of PDA produced increases with increasing conversion. The results convincingly reveal that the polymerization of DA with the CBr₄/Fe(III)/bpy system at 80°C is a living radical polymerization.

The GPC curves of PDA are shown in Figure 4 and M_w/M_n are uniform throughout the polymerization, indicating a controlled polymerization. This observation together with $\ln([M]_0/[M])$ versus time indicates that the initiation is fast and that the contribution of chain breaking, transfer, and termination reactions

Initiation :**Propagation :**

Scheme 1

during the course of polymerization is negligible. MWs determined from GPC are lower than the theoretically predicted value. This disparity may be due to the difference in hydrodynamic volumes between the PDA polymer and the polystyrene standard. Because branched polymers have a lower hydrodynamic volume than linear ones, the polymers will elute later in GPC, leading to apparently lower $M_{n(\text{GPC})}$ values. For comparison of the M_w/M_n free radical polymerization of DA was studied with AIBN as the initiator in presence of bpy at 80°C for 6 h. The $M_{n(\text{GPC})}$ was 2.62×10^4 with a M_w/M_n of 3.3. From these results, we can see that in free radical polymerization, M_w/M_n are much higher than that predicted by ATRP mechanism.

Studies³¹⁻³³ reveal that transition metal complexes play a significant role in vinyl polymerization under usual reaction conditions. The same metal ion can accelerate, retard, and initiate the polymerization reaction. The effect depends upon the method of initiation, the ligand associated or coordinated with the metal ion, and the mechanism of the reaction.³³ They may also induce an atom transfer reaction or provide a source of metal complexed radicals or even initiate a polymerization catalytic cycle that does not involve radical interactions.^{34,35} Therefore, the reduction of R_p

with increasing [Fe(III)] in the polymerization of DA may be due to the retarding effect of Fe(III) complexes. The ATRP mechanism relies on a fast equilibrium between dormant polymeric halide and active species (group radical). CCl_4 is a multifunctional initiator and it is the most widely used halogen in redox telomerization.^{9,36-38} The ability of a $-\text{CCl}_3$ end group to be activated by a metal catalyst in redox telomerization prompted many researchers to use CCl_4 as a promoter of bistelomerization. According to Boutevin et al.³⁸ the lower molecular weight of the polymers with CCl_4 as the initiator may be due to the generation of new polymer chains occurring once the third active site is created per chain. The homogeneous ATRP of DA initiated by CBr_4 with Fe(III)/bpy catalyst system has been proposed to proceed by the same mechanism as that proposed for the CBr_4 initiated ATRP of OMA²⁹ using the Fe(III)/bpy initiator system in the presence of AIBN as the source of reducing agent (Scheme 1).

To gain insight into the nature of the chain ends of low-molecular-weight PDA, the $^1\text{H-NMR}$ analysis of recrystallized PDA is shown in Figure 5 where the important resonance has been assigned. The polymer was prepared at 80°C using AIBN as the radical source and CBr_4 as the initiator in the presence of Fe(III)/bpy

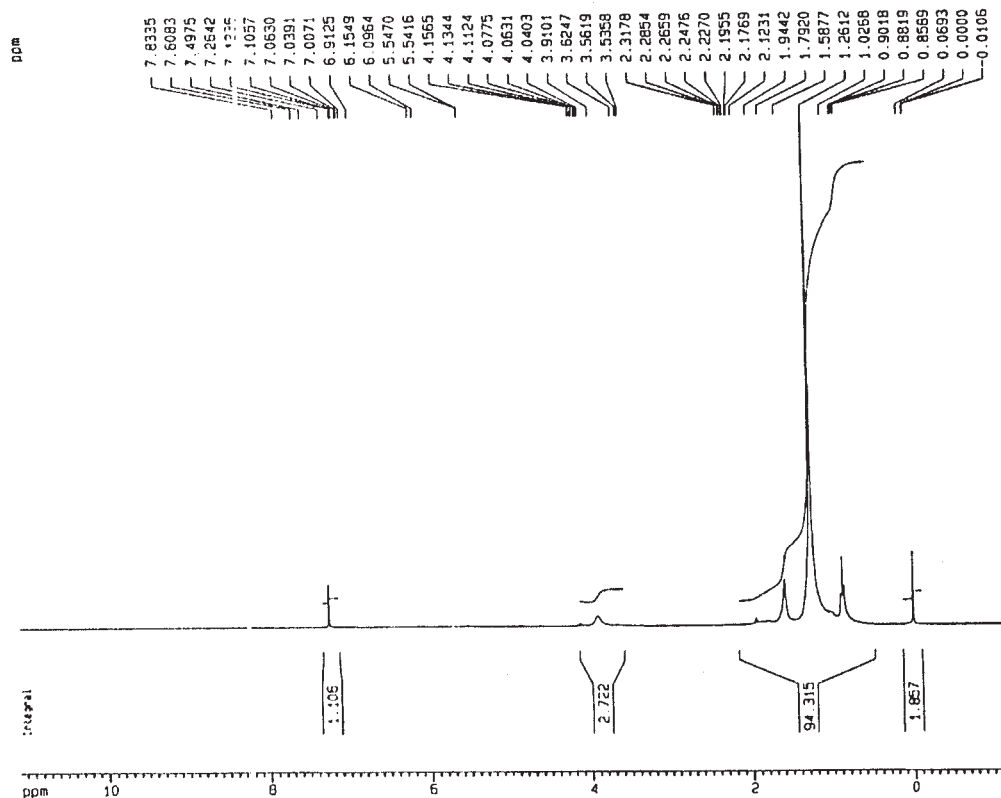
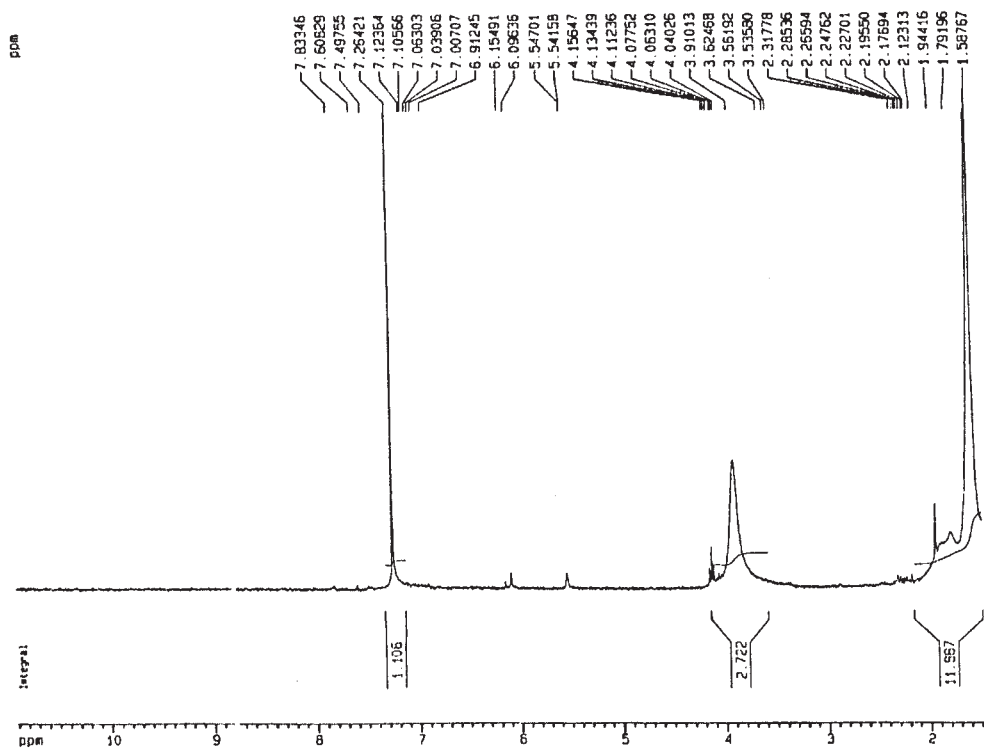


Figure 5 $^1\text{H-NMR}$ of PDA synthesized by ATRP of DA at 80°C in DMF in the presence of AIBN as the source of reducing agent. Conditions: $[\text{DA}] = 1.00 \text{ mol L}^{-1}$; $[\text{AIBN}] = 1.00 \times 10^{-2} \text{ mol L}^{-1}$; $[\text{CBr}_4] = 0.10 \text{ mol L}^{-1}$; $[\text{Fe(III)}] = 1.00 \times 10^{-3} \text{ mol L}^{-1}$; $[\text{bpy}] = 3.00 \times 10^{-3} \text{ mol L}^{-1}$.

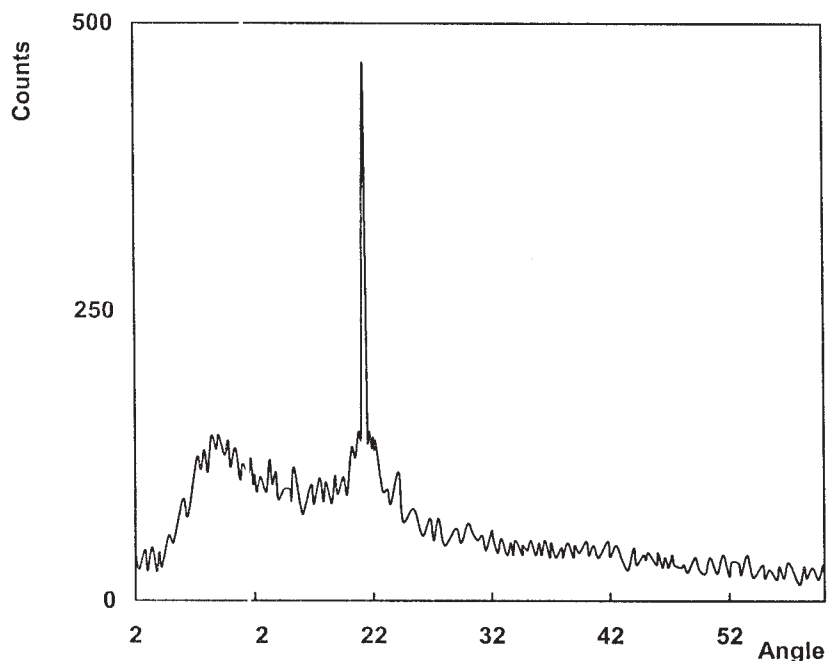


Figure 6 Typical X-ray diffraction pattern of PDA synthesized by ATRP of DA at 80°C in DMF in the presence of AIBN as the source of reducing agent.

catalyst system. Characteristic resonance originating from α -halocarbonyl moieties is visible at both 4.1 and 3.91 ppm. The signal at 4.1 ppm corresponds to the methylene proton in the α -position of the repeated docosyl ester group and the ester group adjacent to the terminal halogen resonates at 3.91 ppm. The protons of $-\text{CH}$ and $-\text{CH}_2$ backbone correspond to reso-

nances at 2.19 and 1.58 ppm, respectively. Similar spectra for PMMA obtained from Fe(III) catalyzed ATRP of MMA were reported in the literature.^{20,39} These results indicate that PDA is end-functionalized with the ω -bromine end group from the catalyst. The M_n calculated from the $^1\text{H-NMR}$ spectrum is not close to that obtained from $M_{n(\text{GPC})}$. This is evident from the

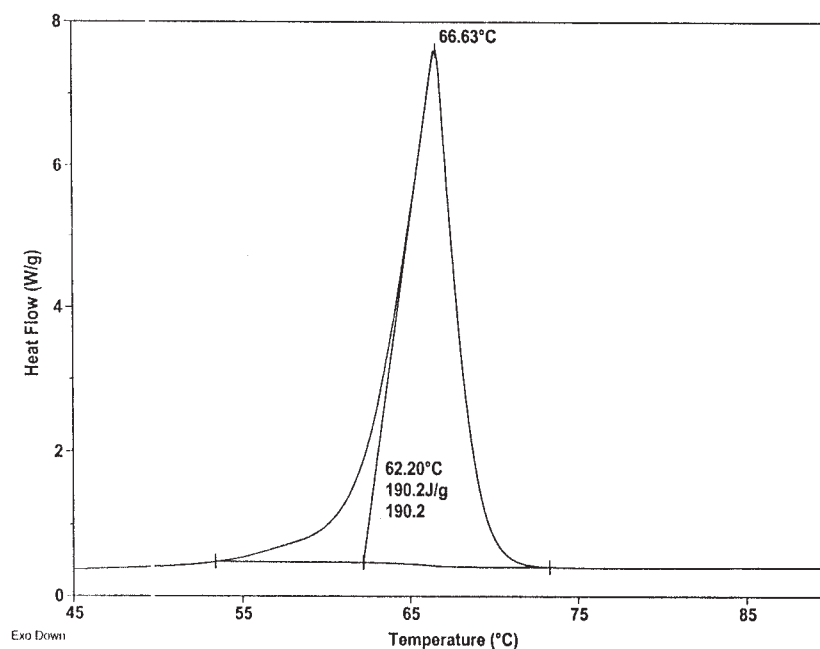


Figure 7 Typical DSC thermogram (scanning rate = 10°C min⁻¹) of PDA synthesized by ATRP of DA at 80°C in DMF in the presence of AIBN as the source of reducing agent.

fact that $M_{n(\text{GPC})}$ of PDA was obtained from a polystyrene standard.

The X-ray diffraction pattern of PDA shows a peak at about $2\theta = 22^\circ$, which corresponds to 4.18-Å spacing (Fig. 6). This corresponds to the value of several long-chain acrylates and methacrylate comb-like polymers,⁴⁰ attributed to the peculiar layered structure for the conventional comb-like polymers, which is made up of several parallel alkyl side chains arranged on both sides of the main chain.⁴¹ PDA prepared by the present initiation system shows a single absorption band at 723 cm^{-1} , indicating that the long alkyl side chains of PDA are of hexagonal type packing.^{41,42}

Figure 7 shows a typical DSC curve of PDA. Melting endothermic peaks were observed at 65–68°C for the PDA samples. The melting points (m_p) and heat of fusion (ΔH_m) were determined from the endothermic peaks. The m_p and ΔH_m values were almost constant regardless of percentage conversion and molecular weights of polymers. The value of entropy of fusion (ΔS_m) was also constant like ΔH_m (Table I), signifying that the m_p changes were not very predominant.

CONCLUSIONS

In this investigation, Fe(III)/bpy catalyzed ATRP of DA has been successfully carried out with CBr_4 as the initiator in the presence of AIBN as the source of the reducing agent. A well-defined PDA with a quite narrow M_w/M_n was obtained through this novel ATRP system. The present work again suggested that the presence of a catalytic amount of growing radicals and a fast equilibrium between growing radicals and dormant species are essential for ATRP of *n*-alkyl (meth)acrylates having pendent alkyl side chains.

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