Journal of Fluorine Chemistry 131 (2010) 616–620

Contents lists available at ScienceDirect

Journal of Fluorine Chemistry

journal homepage: www.elsevier.com/locate/fluor

# Synthesis characterizations and properties of a new fluoro-maleimide polymer

S.M. Mokhtar [*], S.M. Abd-Elaziz, F.A. Gomaa

*Chemistry Department, Faculty of Women Ain-Shams University, Cairo, Egypt*

ARTICLE INFO

*Article history:*
Received 3 October 2009
Received in revised form 29 December 2009
Accepted 6 February 2010
Available online 11 February 2010

*Keywords:*
Fluoro-maleimide polymers
Free radical polymerization
Kinetics
FTIR
NMR
Thermal analysis

ABSTRACT

Novel 4-(4-trifluoromethyl)phenoxy *N*-phenyl-maleimide (FPMI) was synthesized. The free radical-initiated polymerization of FPMI was carried out in 1,4-dioxane solution using azobisisobutyronitrile as initiator. The monomer was investigated by FTIR, $^1$H NMR, $^{13}$C NMR and elemental analysis, while the polymer was investigated by FTIR, $^1$H NMR and $^{13}$C NMR. The effect of the monomer concentration, initiator concentration and temperature on the rate of polymerization ($R_p$) was studied. The activation energy of the polymerization was calculated ($\Delta E = 48.94$ kJ/mol). The molecular weight of PFPMI ($\overline{M}_w$ and $\overline{M}_n$) and polydispersity index of the polymer were determined by gel permeation chromatography and were equal to 73,500, 16,700 and 2.27, respectively. The properties of PFPMI, including thermal behavior, thermal stability, the glass transition temperature ($T_g = 236$ °C), photo-stability, solubility and solution viscosity were studied.

## 1. Introduction

Fluorinated polymers have significant properties such as being highly thermal, chemical, and weather resistance. They have low dielectric constants, refractive index, surface energy and flammability. The presence of fluorine in the side groups of the polymer has a profound effect on almost all the properties of those materials [1,2]. This effect is so striking that fluoropolymers are often treated as separate and remarkable class of polymers with properties unmatched in any other system. Fluorine confers extreme hydrophobicity and water insolubility to polymers. It raises the thermal and oxidative stability. In some instants, the element generates an interchain interaction that is so strong that disables the polymer from dissolving in any solvent. It is the C–F bond that grants such properties, rather than the element itself. The fluoropolymers are biocompatible because of their hydrophobicity [3]. In addition, their excellent inertness to solvents, hydrocarbons, acids, alkalis and moisture adsorption as well as their interesting oil and water repellency, due to the low polarizability, and the strong electronegativity of the fluorine atom make them advantageous [4–13]. Consequently, fluorine-containing polymers have widespread applications in modern technologies ranging from building, automotive and aerospace industries to optics and microelectronics [14–16].

The polymers of *N*-phenyl-maleimide and their derivatives have been known to exhibit high $T_g$s due to the rigid imide rings in the backbones [17,18]. Polymers of N-substituted maleimides and their derivatives, which are classified as polyimides, are one of the important high performance engineering plastics. The presence of a five-member planar ring in the chain hinders the rotation of the imide group around the backbone chain of the macromolecules. This makes them rigid polymers with superior mechanical and thermal stability. The incorporation of fluorine atoms (or groups containing fluorine atoms) into polymer chains increases solubility, glass transition temperature ($T_g$) and thermal stability of polymers in addition to decreasing moisture absorption and dielectric constant. The aromatic fluoropolymers have currently been used as films, coatings and microelectronic devices [19–22]. *N*-Phenyl-maleimide is important in a wide variety of applications. Its derivatives have been suggested as intermediates in the cross-linked reactions of the polyimides [23]. It also serves as an excellent model compound for the important class of resin/fiber advanced materials based on bismalimides [24]. The free radical chain polymerization of 1H-pyrrol-2,5-dione, more commonly referred to as maleimides, and the N-substituted derivatives have been extensively studied [18,25–28].

Many studies have been carried out on the free radical homopolymerization of N-substituted maleimides. The focus has always been on the effect of N-substituted on the reactivity of the monomer/or the final physical and chemical properties of the polymer [17,18,29–34]. The rate of polymerization was found to be influenced by solvent [35–38], initiator [32,37,39], temperature [32,37,25] and additives [30].

In this paper, we have synthesized a new fluoro-maleimide monomer of 4-(4′-trifluoromethyl)phenoxy *N*-phenyl-maleimide

---

[*] Corresponding author at: Chemistry Department, Faculty of Women Ain-Shams University, Asmaa Fahmy St., Heliopeless, Cairo, Egypt. Tel.: +20 106609863.
*E-mail address:* samiamokhtar@hotmail.com (S.M. Mokhtar).

0022-1139/$ – see front matter © 2010 Elsevier B.V. All rights reserved.
doi:10.1016/j.jfluchem.2010.02.003

[FPMI] that is readily polymerized by free radical mechanism using 2,2′-azobisisobutyronitrile as initiator in 1,4-dioxane.

## 2. Experimental

### 2.1. Analytical and spectroscopic methods

The FTIR spectra of the prepared monomer and its polymer were recorded by IR spectroscopy (8201 Pc Shimadzu) and Varian 200 MHz. $^1$H and $^{13}$C NMR spectra were recorded in $CDCl_3$ for monomer and DMSO for polymer with tetramethylsilane as internal standard on Varian Mercury VX-500 NMR spectrometer. NMR spectra were run at 500 MHz and for $^{13}$C NMR 125 MHz. The X-ray analysis carried out by X-ray diffraction instrument Philips Pw 1390 channel control. Cu target $K\alpha = 1.542$, Ni filter is 40 kV, 25 mA. The viscosity measurements were carried out using an Ubbelohde viscometer suspended level dilution viscometer. Dioxane was used as solvent with a flow time of 121 s at 25 °C. The gel permeation chromatography (GPC) of the samples was performed using 1100 Aligant instrument equipped with organic GPC-SEC start up kits with a flow rate of 1 ml/min, THF as mobile phase, maximum pressure 150 bar, minimum pressure 5 bar, injection volume 20 μl and column temperature thermostat 25 °C. The eluent was monitored with a refractive index detector of optical unit temperature 25 °C and peak width 0.1 min. Polymer concentration was 0.1 wt.%. The thermogravimetric analysis of the PFPMI powder was carried out in nitrogen atmosphere with a heating rate ranging from 10 °C min$^{-1}$ up to 700 °C by Shimadzu TGA-50 H. The glass transition ($T_g$) temperature was investigated by differential scanning calorimeter analysis (DSC). The glass transition ($T_g$) temperature was determined by Shimadzu DSC-50 with a heating rate of 10 °C min$^{-1}$ under nitrogen atmosphere.

### 2.2. Materials

4-Chlorobenzotrifluoride (Fluka), 4-aminophenol (Aldrich), maleic anhydride were purified by recrystallization in ether (m.p. 54 °C), and azobisisobutyronitrile (AIBN) (E. Merck) was recrystallized twice from ethanol (m.p. 104 °C). The other reagents were used as received and the solvents were purified according to conventional methods.

### 2.3. Monomer synthesis

#### 2.3.1. 4-(4-Trifluoromethyl)phenoxy aniline: [22]

In a 500 ml round bottom flask, 24.0 g (0.22 mol) of 4-aminophenol, 14.0 g (0.25 mol) of KOH, 100 ml of toluene and 200 ml of DMSO were added. The mixture was heated to 140 °C with stirring under nitrogen for 3 h to remove produced water by azeotropic distillation with toluene. After complete removal of water, the residual toluene was distilled off. Then, the solution was cooled to 100 °C and 28 g (0.15 mol) of 4-chlorobenzotrifluoride in 40 ml of DMSO was added drop wise. The reaction was carried out with stirring at 120 °C for 8 h. After that, the mixture was poured into 1000 ml of cold water to give pale brown crystals. The precipitate was collected and washed thoroughly with water to give a crude product. Then, it was purified by recrystallization in ethanol/water. Yield 80%. m.p. 78 °C. Anal. Calc. for $C_{13}H_{10}F_3NO$: C, 61.5; H, 3.98; N, 5.53. Found: C, 62.16; H, 4.31; N, 5.52.

#### 2.3.2. 4-(4-Trifluoromethyl)phenoxy N-phenyl-maleimide (FPMI)

FPMI was prepared in two stage procedures originally developed by Searle [40]. In the first stage, 4-(4-trifluoromethyl)phenoxy aniline (0.3 mol) in ether or chloroform was added drop wise with vigorous stirring to a cold solution (10–15 °C) of maleic anhydride (0.3 mol). The resulting product, usually fine precipitate, was filtered, washed with chloroform and dried in an air oven at 60 °C. In the second stage, the obtained maleimic acid was treated with fused sodium acetate (0.614 mol) and acetic anhydride (10 mol) at 70–80 °C for 1 h with occasional shaking and then left at room temperature for 1 day. The reaction products were poured in an ice/water mixture and the maleimide was immediately separated as a light brown crystals. This precipitate was filtered and washed with water till acid free. The crude product was then recrystallized several times from ethanol/water. The fluoromaleimide that was prepared had yield 80%, m.p. 136 °C.

Anal. Calc. for $C_{17}H_{10}F_3O_3N$: C, 61.2; H, 3.0; N, 3.9. Found: C, 62.91; H, 4.4; N 4.2. IR (KBr): $\nu$ 1709 (CO), $\nu$ 1160 (COC), $\nu$ 1603 (NCO), 1400 (C=C), 1326 (CF), 838 (CH) cm$^{-1}$. $^1$H NMR (500 MHz, $CDCl_3$): $\delta$ 7.5 (d, 2H), 7.3 (d, 2H), 7.1 (d, 2H), 7.08 (d, 2H) 6.8 (s, CH=2H). $^{13}$C NMR (125 MHz $CDCl_3$): $\delta$ 169.4 (CO), 159.8, 155.3 (COC), 134.2 (ethylenic, CH), 127.8, 127.2 ($CF_3$), 125.3 (2CH, Ar), 120.1 (2CH, Ar), 118.4 (2H, Ar) (Schemes 1 and 2).

### 2.4. Polymerization procedure

Polymerization was carried out in calibrated dilatometers (3–5 ml in capacity) with ground joint stoppers. The dilatometer contains the required amount of monomer and initiator dissolving in the appropriate amount of solvent was placed in ultra-thermostat. The temperature was regulated to (±0.1 °C). After the required time, the dilatometer was immersed in ice water–salt mixture. The dilatometer was then connected to a vacuum pump and the solution was withdrawn and precipitated in ethanol. The precipitate was filtered and dried at 60 °C to constant weight. The % conversion was determined gravimetrically. The polymers were usually purified by precipitation from ethanol solution into dioxane. The rates of polymerization were determined from the slope of the linear concentration vs. time plots.

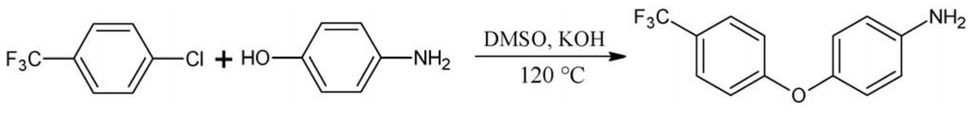
#### 2.4.1. Characterization of PFPMI

IR (KBr): $\nu$ 1708 (CO), $\nu$ 1168 (COC), $\nu$ 1603 (NCO), 1328 (CF), 2971, 838 (CH) cm$^{-1}$. $^1$H NMR (DMSO): $\delta$ 7.7 (d, 2H), 7.3 (d, 2H), 7.4 (d, 2H), 7.1 (d, 2H), 6.9 (d, 2H), 3.5–4.5 (s, CH– 2H). $^{13}$C NMR (DMSO): $\delta$ 170 (CO), 159.8, 155.3 (COC), 129.3, 128.1 ($CF_3$), 120.6 (4CH, Ar), 118.9 (4CH, Ar), 40.2–39.8 (CH–CH).

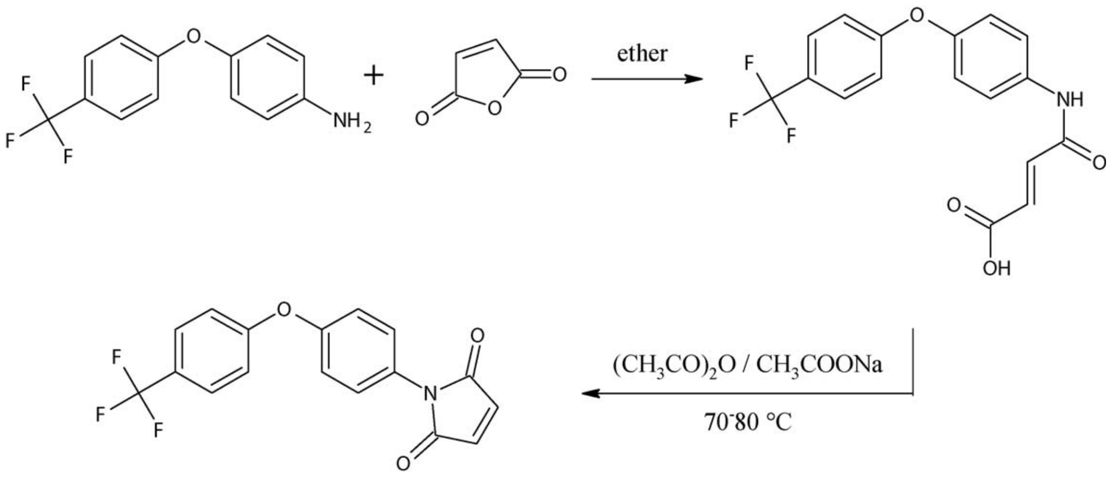
## 3. Results and discussion

The polymerization of FPMI was carried out in the presence of azobisisobuteronitrile as a radical initiator and in dioxane as a solvent. The polymerization process is confirmed by spectral tools.

The FTIR spectra of PFPMI support the polymerization process. The disappearance of the band at 1400 cm$^{-1}$ which is due to the stretching vibration C=C, indicating that the double bond had been disappeared. This means that the polymerization has occurred by the usual free radical addition mechanism. $^1$H NMR of monomer shows band at 6.9 ppm corresponding to the ethylene proton of the double bond and four bands due to the four non-equivalent protons



**Scheme 1.** Synthesis of 4-(4-trifluoromethyl)phenoxy aniline.



**Scheme 2.** Synthesis of 4-(4-trifluoromethyl)phenoxy *N*-phenyl-maleimide (FPMI).

of two phenyl groups. The bands are sharp and narrow characteristic of crystalline monomer. The NMR spectrum of the polymer shows broad bands characteristic of the polymeric material. The band at 6.9 ppm disappeared indicating the disappearance of the double bond. The ethylenic protons of the main polymeric chain appear at 3.5–4.5 ppm which is another indication that polymerization has occurred. $^{13}$C NMR spectra of PFPMI assignments of the peaks are in good agreement with the proposed structure and the polymerization process. The disappearance of the band at 134 ppm of C=C and the appearance of the band at 40.1–39.8 ppm indicate the formation of the saturated bond. This is considered another observation proving the occurrence of polymerization.

The rates of free radical polymerization were measured dilatometrically. All the polymerization proceeded homogeneously.

### 3.1. Dependence of the rate of polymerization on initiator concentration

The effect of the initiator on the rate of free radical polymerization was studied at fixed monomer concentration of 0.75 mol/l at 77 °C. The concentration of the initiator was varied and log initial rate of polymerization was plotted vs. log initiation concentration. A straight line was obtained as shown in Fig. 1 the slope of the line gives the kinetic order with respect to the initiator and is equal to 0.45. From this value, the rate of polymerization is proportional with the limits of error to initiator concentration ($I^{0.5}$). This means that the dependence of $R_p$ on $[I]$ is a direct consequence of the bimolecular termination mechanism.

### 3.2. Dependence of the rate of polymerization on the monomer concentration

The dependence of the initial rates of polymerization ($R_p$) of FPMI on the monomer is shown in Fig. 2, which is a log plot of the rate vs. monomer concentration at constant initiator concentration $[I]$, $2 \times 10^{-2}$ mol/l, and at temperature of 77 °C. The rate of polymerization $R_p$ has been determined from the slope of the change in volume ($\Delta v$) vs. time. The slope of the log plot of the rate vs. monomer concentration equals to 1.1. This value represents the order of the reaction regarding the monomer concentration. It is a little bit higher than the order of magnitude, which is unity characteristic to free radical polymerization. This may be attributed to steric and solvent effects. Finally, the steady state of polymerization is:

$$R_p = k_p\left(\frac{k_d}{k_t}\right)^{0.5}[I]^{0.45}[M]$$

where $k_p$, $k_d$ and $k_t$ are the rate constants of propagation, initiator decomposition and termination, respectively.

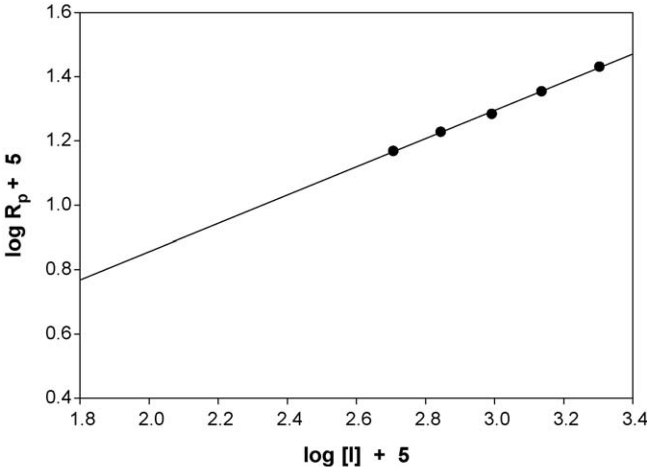


**Fig. 1.** Effect of initiator concentration on the rate of polymerization in dioxane, the monomer concentration $[M]$ = 0.75 mol/l and at 77 °C.

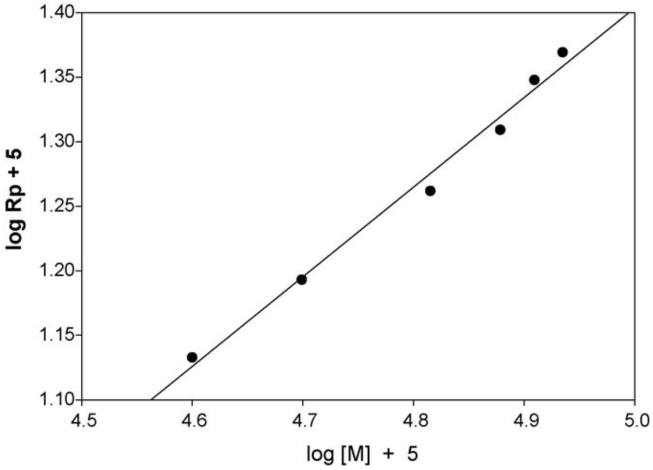


**Fig. 2.** Effect of monomer concentration on the rate of polymerization in dioxane, the initiator concentration $[I] = 2 \times 10^{-2}$ mol/l and at 77 °C.

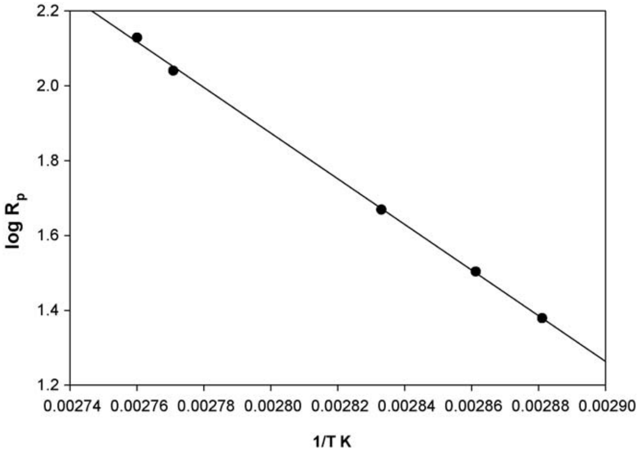


**Fig. 3.** Effect of temperature on the rate of polymerization in dioxane, $[M] = 0.75$ mol/l and $[I] = 2 \times 10^{-2}$ mol/l.

The data indicate that the rate of polymerization should vary with the square root of the initiator concentration and with the first power of the monomer concentration according to the normal reaction of free radical polymerization mechanism.

### 3.3. Effect of temperature

The effect of temperature on the rate and degree of polymerization is of prime importance in determining the manner of performing a polymerization. Increasing the reaction temperature usually increases the polymerization rate and decreases the polymer molecular weight. Fig. 3 shows the effect of temperature on the rate of polymerization. The activation energy of the polymerization can be calculated or expressed by Arrhenius-type equation:

$$K = Ae^{-E/RT}$$

where $A$ is the collision frequency factor, $R$ is the gas constant, $T$ is the Kelvin temperature, and $E$ is the activation energy ($E = 1/2E_{\mathrm{d}} + E_{\mathrm{p}} - 1/2E_{\mathrm{t}}$), where $E_{\mathrm{d}}$, $E_{\mathrm{p}}$ and $E_{\mathrm{t}}$ are the activation energies of the rate of initiator decomposition, propagation and termination, respectively. The activation energy ($E$) was calculated from the slope of plot log $k$ vs. $1/T$ and was found to be equal to 48.94 kJ/mol. This value was found in the range in most cases of free radical polymerization [41].

### 3.4. Molecular weight determination

The intrinsic viscosity of the prepared polymer was measured in dioxane at 25 °C using Ubbelohde viscometer. Fig. 4 shows that the intrinsic viscosity $[\eta]$ increases with increasing monomer concentration and decreases with increasing the initiator concentration, which is a well expected behavior for free radical polymerization. The weight average ($\overline{M}_w$) and number average ($\overline{M}_n$) molecular weight and polydispersity index ($\overline{M}_w/\overline{M}_n$) of polymer sample were obtained from gel permeation chromatography. The values of number average and weight average molecular weight of PFPMI are equal to 73,500 and 16,700, respectively. The polydispersity index of the PFPMI is equal to 2.27. These data indicate the prepared polymer has a high molecular weight.

### 3.5. Characterization

#### 3.5.1. X-ray diffraction

The X-ray method allows the calculation of the relative amounts of crystalline and amorphous nature in a polymer. The crystallinity of PFPMI polymer was examined by X-ray diffraction. The X-ray diffraction exhibits amorphous patterns [42]. These results could be explained by the presence of bulky pendant groups, which disrupted the regularity of the molecular chains and inhibited the close packing of the polymer chains.

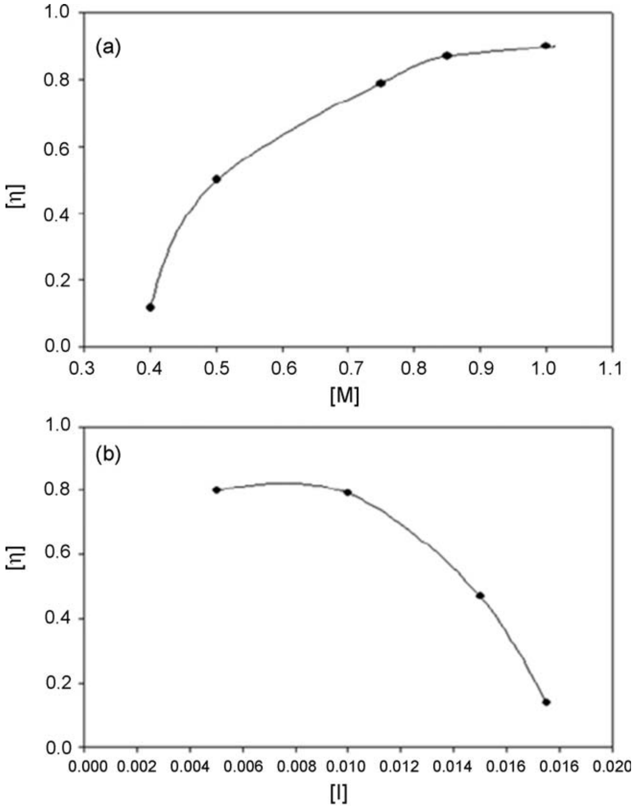


**Fig. 4.** Effect of monomer concentration and initiator concentration on the intrinsic viscosity.

**Table 1**
Solubility of poly 4-(4′-trifluoromethyl)phenoxy *N*-phenyl-maliemide.

| Solvent | Solubility |
|---|---|
| Acetic acid | − |
| Choroform | + |
| DMF | + |
| DMSO | + |
| THF | + |
| Actone | + |
| Methanol | − |
| Ethanol | − |
| Diethylether | − |
| Benezene | − |
| Petroleumether | − |
| Carbontetrachloride | − |

−, insoluble; +, soluble.

#### 3.5.2. Solubility

Table 1 shows the solubility of the new PFPMI towards some organic solvent and organic acid. The data indicate that PFPMIs are soluble in some organic solvents including non-polar solvents. The solubility of PFPMI could be attributed to the presence of bulky pendant groups, which led to disturbing the regularity and crystallinity of molecular chains and increased the free volume. The resistance of the new polymer to other solvents may be due to the polar nature of the trifluoromethyl group.

#### 3.5.3. Thermal properties

Thermogravimetric analysis was used to evaluate the thermal stability of PFPMI. The initial decomposition temperature ($T_{\mathrm{init}}$)

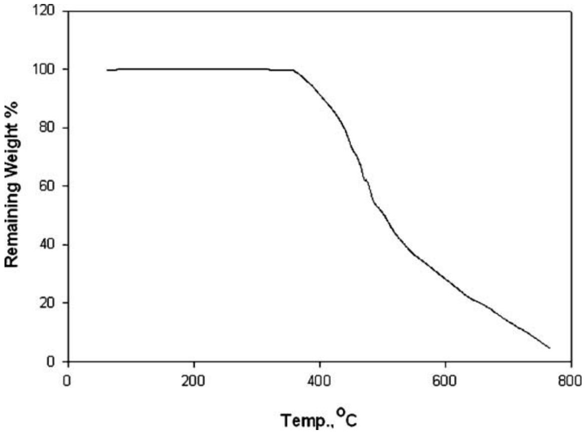


**Fig. 5.** Thermogravimertic analysis of PFPMI.

and the maximum decomposition temperature ($T_{\max}$) were determined by thermogravimetric analysis. Fig. 5 shows the thermogram for PFPMI in nitrogen at a heating rate of 10 °C min$^{-1}$. The diagram shows that the initial decomposition temperature ($T_{\text{init}}$) is equal to 384.72 °C with 10.39% weight loss, the maximum decomposition temperature ($T_{\max}$) is equal to 585 °C with 28% weight.

The data indicate that the PFPMI has an excellent thermal stability as well as the other fluoro and maleimide polymers.

In addition, the glass transition ($T_g$) was determined by DSC. The glass transition temperature ($T_g$) of PFMI was observed to be about 236 °C.

PFPMI showed good thermal and photo-stability. The polymer did not show any changes in appearance when it was heated in air up to 400 °C. Furthermore, it did not show any change in appearance or weight loss when it was exposed to the UV lamb of long and short wave for an interval time of 10 days. The FTIR spectra of the heated samples or the exposed samples showed no changes in the position of the characteristic peaks. This means that the structure of the treated sample did not change.

From the above, we can conclude that PFPMI have an excellent thermal and photo-stability.

## 4. Conclusion

The new trifluoromethyl phenoxy *N*-phenyl-maliemide monomer was synthesized and characterized. The free radical polymerization of FPMI was initiated by azobisisobutyronitrile in 1,4-dioxane. The characterizations of the polymer as well as the rates of free radical polymerization were studied. The activation energy $\Delta E$ of the polymerization was calculated and it is found to be equal to 48.94 kJ/mol.

The FPMI polymer has a high molecular weight. The average molecular weight ($\overline{M}_w$ and $\overline{M}_n$) and polydispersity index are equal to 73,500, 16,700 and 2.27, respectively. The PFPMI bosses high glass transition temperature ($T_g$ = 236). Also, the polymer exhibits a good solubility and high thermal stability as well as photo-stability.

## References

[1] J.M. Barrales-Rienda, J. González Ramos, M. Sánchez Chaves, J. Polym. Sci., Polym. Chem. 17 (1979) 81–96.
[2] A. Matsumoto, K. Takeshi, J. Appl. Polym. Sci. 68 (1998) 1703–1708.
[3] H.R. Allcock, F.W. Lampe, Contemporary Polymer Chemistry, 2nd ed., Hall Inc., Englewood Cliffs, NJ, 1990, 546 pp..
[4] H.M. Wei, V.R. Luc, V.D.G. Robert, Polym. Bull. 47 (2001) 321–328.
[5] I.J. Park, S.B. Lee, C.K. Choi, Polymer 38 (1997) 2523–2527.
[6] B. Ameduri, R. Bongiovanni, G. Malucelli, A. Pollicino, A. Priola, J. Polym. Sci. Part A: Polym. Chem. 37 (1999) 77–87.
[7] V. Pomes, A. Fernandez, N. Costarramone, B. Grano, D. Houi, Colloids Surf. A: Physicochem. Eng. Aspects 159 (1999) 481–490.
[8] S. Yang, J. Wang, K. Ogrino, S. Valiyaveettil, C.K. Ober, Chem. Mater. 12 (2000) 33–40.
[9] J.R. Lee, F.L. Jin, S.J. Park, J.M. Park, Surf. Coat. Technol. 180 (2004) 650–654.
[10] L. Van Ravenstein, W. Ming, R.D. Van de Grampel, R. Van der Linde, G. deWith, T. Loontjens, P.C. Thune, J.W. Niemantsverdriet, Macromolecules 37 (2004) 408–413.
[11] J.R. Fried, Polymer Science and Technology, 2nd ed., Hall Lim, New Delhi, 2005, pp. 402–404.
[12] Z. Ge, X.Y. Zhang, J.B. Dai, W.H. Li, Y.J. Luo, Eur. Polym. J. 45 (2009) 530–536.
[13] I. Erol, J. Fluorine Chem. 129 (2008) 613–620.
[14] E. Sacher, Prog. Surf. Sci. 47 (1994) 273–300.
[15] A. Ghosh, S. Banerjee, H. Komber, K. Schneider, L. Häußler, B. Voit, Eur. Polym. J. 45 (2009) 1561–1569.
[16] F.R. Pu, R.L. Williams, T.K. Markkula, J.A. Hunt, Biomaterials 23 (2002) 2411–2428.
[17] A. Matsumoto, T. Kubota, T. Otsu, Macromolecules 23 (1990) 4508–4513.
[18] T. Otsu, A. Matsumoto, T. Kubota, S. Mori, Polym. Bull. 23 (1990) 43–50.
[19] B.J. Liu, G.B. Wang, W. Hu, Y.H. Jin, C.H. Chen, Z.H. Jiang, W.J. Zhang, Z.W. Wu, Y. Wei, J. Polym. Sci. Part A: Polym. Chem. 40 (2002) 3392–3398.
[20] R.H. Vora, R.S.G. Krishnan, S.H. Goh, T.S. Chung, Adv. Funct. Mater. 11 (2001) 361–373.
[21] V. Kute, S. Banerjee, Macromol. Chem. Phys. 204 (2003) 2105–2112.
[22] Y. Niu, X. Zhu, L. Liu, Y. Zhang, G. Wang, Z. Jiang, React. Funct. Polym. 66 (2006) 559–566.
[23] T.T. Serafini, P. Delvigs, G.R. Lightsey, J. Appl. Polym. Sci. 16 (1972) 905–915.
[24] B.A. Rozenberg, G.N. Boiko, R.J. Morgan, E.E. Shin, Polym. Sci. Ser. A 43 (2001) 386–399.
[25] S. Iwatsuki, M. Kubo, M. Wakita, Y. Matsui, H. Kanoh, Macromolecules 24 (1991) 5009–5014.
[26] A. Matsumoto, Y. Oki, T. Otsu, Eur. Polym. J. 29 (1993) 1225–1229.
[27] T. Oishi, H. Yamaski, M. Fujimoto, J. Polym. 23 (1991) 795–804.
[28] T. Sato, K. Masaki, K. Kondo, M. Seno, H. Tanaka, Polym. Bull. 35 (1995) 345–350.
[29] T. Oishi, M. Fujimoto, J. Polym. Sci., Polym. Chem. 22 (1984) 2789–2800.
[30] M.Z. El Sabee, S. Mokhtar, Eur. Polym. J. 19 (1983) 451–456.
[31] J. Lokaj, F. Hrabak, Eur. Polym. J. 14 (1978) 1039–1043.
[32] J.E. Wang, Y.T. Chern, M.A. Chung, J. Polym. Sci. Part A: Polym. Chem. 34 (1996) 3345–3354.
[33] A. Matsumoto, T. Kimura, J. Macromol. Sci. Pure Appl. Chem. Part A 33 (1996) 1049–1061.
[34] S.F. Parker, Spectrochim. Acta Part A: Mol. Biomol. Spectrosc. 51 (1995) 2067–2072.
[35] H. Aida, I. Takase, T. Nozi, Macromol. Chem. Phys. 190 (1989) 2821–2831.
[36] K. Qiu, K. Ye, J. Acta Polym. Sin. 1 (1993) 125–128 (in Chinese).
[37] A. Matsumoto, T. Kubota, H. Ito, T. Otsu, J. Mem. Fac. Eng. Img., Osaka City Univ. 31 (1990) 47–59.
[38] T. Oishi, K. Sase, K. Saeki, S. Yao, K. Ohdan, Polymer 36 (1997) 3935–3942.
[39] M. Yamada, I. Takase, N. Koutou, J. Polym. Sci. B, Polym. Lett. 6 (1968) 883–888.
[40] N.E. Searle (Inventor), US Patent, 2,444,356 (1948).
[41] P. Ghosh, Polymer Science and Technology Plastics, Rubbers, Blends and Composites, 2nd ed., Tata McGraw-Hill, 2002.
[42] P.C. Dawson, D.J. Blundell, Polymer 21 (1980) 577–578.