

# Physics and Kinetics of Alkaline Hydrolysis of Cationic Dyeable Poly(ethylene terephthalate) (CDPET) and Polyethylene Glycol (PEG)-Modified CDPET Polymers: Effects of Dimethyl 5-Sulfoisophthalate Sodium Salt/PEG Content and the Number-Average Molecular Weight of the PEG

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**ABSTRACT:** This study investigated the physics and kinetics of three cationic dyeable poly(ethylene terephthalate) (CDPET) and four polyethylene glycol (PEG)-modified cationic dyeable PET (PEG-modified CDPET) polymers using differential scanning calorimetry, thermogravimetric analysis, the density gradient method, gel permeation chromatography, a rheometer, and statistical regression analysis. The molar ratios of dimethyl 5-sulfoisophthalate sodium salt (SIPM) for three CDPET polymers were 2, 6, and 10%, respectively. For four PEG-modified CDPET polymers, with molar ratios of SIPM of 2%, the PEG content and the number-average molecular weights ( $M_n$ ) of PEG were 3/6 (wt %) and 400/2000 (g/mol), respectively. Experimental results revealed that the crystallinities of CDPET and PEG-modified CDPET polymers declined as the SIPM and PEG contents increased. The crystallinity of the PEG400-CDPET polymer exceeded that of the PEG2000-CDPET polymer with the same SIPM and PEG contents. The SIPM and PEG segments

did not change the original PET unit cell. The  $-\text{SO}_3\text{Na}$  functional group and the PEG molecules gathered only on the crystal surface and in the amorphous regions. In the kinetic equation of the alkaline hydrolysis of PET, CDPET, and PEG-modified CDPET polymers,  $\beta$  equals 1. Furthermore, the  $R^2$  of the kinetic equation for  $\alpha$  from 1.09 to 1.25 was better than that of  $\alpha = 1$ . The rate constants of alkaline hydrolysis were in the order CDPET-10 > CDPET-6 > PEG2000-6-CDPET-2 > PEG2000-3-CDPET-2 > PEG400-6-CDPET-2 > PEG400-3-CDPET-2 > CDPET-2 > PET. Clearly, the rate constant of the alkaline hydrolysis of CDPET and PEG-modified CDPET polymers increased with SIPM/PEG content and the  $M_n$  of the PEG increased. © 2005 Wiley Periodicals, Inc. *J Appl Polym Sci* 98: 550–556, 2005

**Key words:** density; kinetics (polym.); crystal structures; CDPET; statistical regression analysis

## INTRODUCTION

Poly(ethylene terephthalate) (PET) is a useful fiber because it is strong, inert, and thermally stable. These properties also favor its use in microelectronics and solid-state devices. The polyester has a regular linear structure characterized by repeated units that form the backbone of the molecular chain.<sup>1</sup> Hall and Whinfield<sup>2</sup> were the first to successfully treat PET with aqueous sodium hydroxide (NaOH) solution to improve the handling of the fiber and fabric. Latta et al.<sup>3,4</sup> also found that treatment with alkali improved the absorption of moisture, as well as the antistatic and soil-release properties of the fiber.

Some reports have addressed the kinetics of alkaline hydrolysis, as follows. Namboori<sup>5</sup> found that treating PET with different concentrations of aqueous NaOH solution would cause the initial concentration of NaOH to have a substantial effect on the weight loss of the fiber. At a constant temperature, the weight loss was proportional to the time of hydrolysis. Although this observation was consistent with that of Waters,<sup>6</sup> Pficer<sup>7</sup> posited that the weight lost by PET during alkaline hydrolysis varied exponentially with the time of treatment. In an investigation of the alkaline hydrolysis of PET fiber, Kallay et al.<sup>8</sup> stated that at a constant concentration of aqueous NaOH solution, the weight loss was proportional to a specific area of the fiber. Heidemann<sup>9</sup> noted that the radius of the fiber decreased as the concentration of aqueous NaOH solution increased. In light of these developments, this work thoroughly investigates the crystallinity, thermal behavior, flow behavior, and kinetic behavior as-

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TABLE I  
Synthetic Characteristics of PET, CDPET, and PEG-Modified CDPET Chips<sup>a,b</sup>

Code	Composition	PEG (wt %)	SIPM (mol %)	$\bar{M}_n$ of the PEG (g/mol)	I.V. (dL/g)	$\bar{M}_w/\bar{M}_n$	$\bar{M}_w/\bar{M}_n$	DEG (%)	R-COOH (meq/kg)	$T_m$ (°C)	$T_d^c$ (°C)
Sample 1	PET	—	0	—	0.64	39,000	3.56	1.04	32.4	254	396
Sample 2	CDPET-2	—	2	—	0.50	33,000	2.39	2.10	55.3	248	383
Sample 3	PEG400-3-CDPET-2	3	2	400 ± 50	0.49	31,000	2.34	2.51	50.1	246	380
Sample 4	PEG400-6-CDPET-2	6	2	400 ± 50	0.48	30,000	2.19	2.83	47.6	243	375
Sample 5	PEG200-3-CDPET-2	3	2	2000 ± 100	0.47	29,000	2.46	2.72	54.8	242	377
Sample 6	PEG2000-6-CDPET-2	6	2	2000 ± 100	0.46	28,000	2.37	2.97	58.2	240	376
Sample 7	CDPET-6	—	6	—	0.45	27,000	2.27	3.75	46.2	230	374
Sample 8	CDPET-10	—	10	—	0.39	21,000	2.14	7.56	40.4	202	370

<sup>a</sup> Including CDPET-2, CDPET-6, and CDPET-10 polymers.

<sup>b</sup> Including PEG400-3-CDPET-2, PEG400-6-CDPET-2, PEG2000-3-CDPET-2, and PEG2000-6-CDPET-2 polymers.

<sup>c</sup> Degradation temperature.

sociated with the alkaline hydrolysis of cationic dyeable CDPET and polyethylene glycol (PEG)-modified CDPET polymers. The effects of dimethyl 5-sulfoisophthalate sodium salt (SIPM)/PEG content and the number-average molecular weight ( $\bar{M}_n$ ) of the PEG are considered.

## EXPERIMENTAL

### Materials

Regular PET was kindly donated by the Far Eastern Textile Ltd. in Taiwan. Three CDPET and four PEG-modified CDPET polymers were obtained from the Van Nung Institute of Technology in Taiwan. Table I presents the synthetic characteristics of PET, three CDPET, and four PEG-modified CDPET chips.

### Measurements

Differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA) of samples were performed using a Perkin-Elmer Pyrix-1 apparatus (Perkin-Elmer Cetus Instruments, Norwalk, CT). The heating and cooling rates were 10°C/min from 30 to 300 and 300 to 30°C, respectively, and the temperature was maintained for 3 min at 300°C for DSC measurement. The heating rate used for the TGA measurement was 10°C/min from 30 to 600°C. The crystallinity obtained by the DSC method was calculated using the following equation: crystallinity (%) =  $\Delta H_m/\Delta H_m^0 \times 100$  (%), where  $\Delta H_m$  and  $\Delta H_m^0$  are the heat of fusion in J/g of the repeating unit in the samples and the heat of fusion at 100% crystallinity, respectively.  $\Delta H_m^0$  of PET is 140.1 J/g.<sup>10</sup> The density of samples was measured at 25°C by use of the density gradient method, using toluene-carbon tetrachloride mixtures. The crystallinity obtained by the density method was determined using the following equation: crystallinity

(%) =  $d_c(d - d_a)/d(d_c - d_a) \times 100$  (%), where  $d_c$  is the density of the crystal and  $d_a$  is the density of the amorphous phase; these values were taken as 1.445 and 1.335 g/cm<sup>3</sup>, respectively.<sup>11,12</sup> The viscosity of the melt was measured by a capillary rheometer; the capillary length-to-diameter ratio was 5. All stress-strain data of samples were obtained using a Zwick 1511 type Instron instrument (Zwick, Bamberg, Germany) at an extension rate of 10 mm/min. The specimen was 50 mm long, 10 mm wide, and 0.5 mm thick. The increasing value of the tensile strength of the specimen was recorded on a moving chart. The measured weight lost by the samples after treatment with alkali was calculated as follows: weight loss (%) =  $(W_1 - W_2)/W_1 \times 100$  (%), where  $W_1$  and  $W_2$  are the weight of the samples before and after alkaline hydrolysis, respectively.<sup>13</sup> The specific area of the samples was measured using a Flowsorb II 2300 specific area meter (MicroMetrics, Inc., Londonderry, NH).

## RESULTS AND DISCUSSION

### Analysis of crystallinity

The crystallinities of PET, CDPET, and PEG-modified CDPET materials were determined by applying the density gradient method. The experimental results in Table II show that the crystallinity of the CDPET polymer declined as the SIPM content increased. This tendency was consistent with the results of Teli and Prasad<sup>14</sup> and Pal et al.<sup>15</sup> for anionic polymers. However, the crystallinity of the PEG-modified CDPET polymer decreased as the PEG content increased, given a fixed SIPM content. At the same SIPM and PEG contents, the crystallinity of the PEG400-CDPET polymer slightly exceeded that of the PEG2000-CDPET polymer. In the DSC method, the heat of fusion was used to indicate the crystalline fraction of the material. A higher value of heat of fusion was ex-

**TABLE II**  
Crystallinities of PET, CDPET, and PEG-Modified CDPET Chips

Code	Density gradient method		DSC method	
	$d$ (g/cm <sup>3</sup> )	$X_c$ (%)	$\Delta H_m$ (J/g)	$X_c$ (%)
Sample 1	1.3756	35.8	43.8	31.3
Sample 2	1.3717	32.2	42.2	30.1
Sample 3	1.3707	31.4	40.9	29.2
Sample 4	1.3695	30.3	39.3	28.1
Sample 5	1.3682	29.2	38.6	27.6
Sample 6	1.3667	27.9	36.5	26.1
Sample 7	1.3572	19.7	24.4	17.4
Sample 8	1.3426	6.8	6.6	4.7

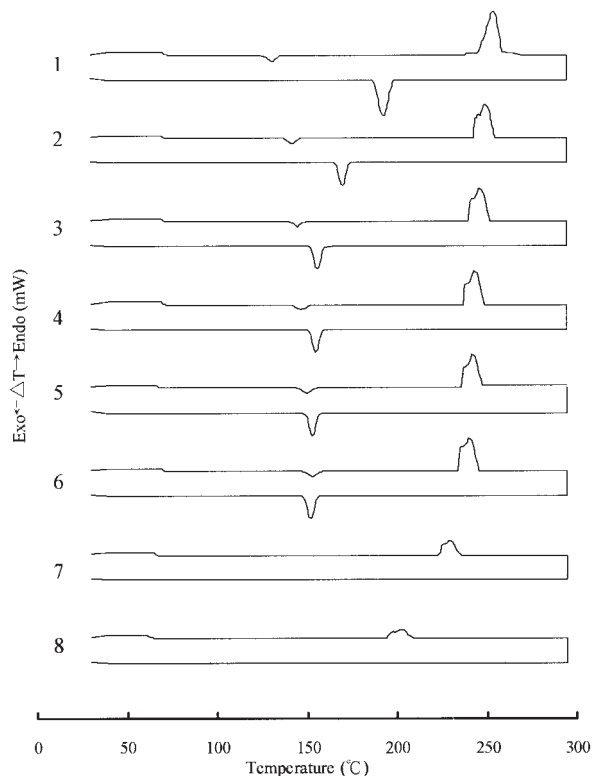
pected to induce a higher crystallinity. Notably, increasing the SIPM and PEG contents in the CDPET and PEG-modified CDPET polymers reduced the heat of fusion, thus decreasing the crystallinity. The crystallinity of the CDPET polymer exceeded that of the PEG-modified CDPET polymer for a fixed SIPM content. For specific SIPM and PEG contents, the crystallinity of the PEG400–CDPET polymer exceeded that of the PEG2000–CDPET polymer; that obtained by the DSC method was consistent with that obtained by the density gradient method. The crystallinity obtained using the DSC method was slightly less than that obtained using the density gradient method.

### Analysis of thermal behavior

Table III and Figure 1 present the thermal behaviors of PET, CDPET, and PEG-modified CDPET polymers. The melting endothermic peak ( $T_m$ ) of the DSC curve in the heating process was different. A double melting endothermic peak was obtained from CDPET and PEG-modified CDPET polymers and only a single

**TABLE III**  
Thermal Properties of PET, CDPET, and PEG-Modified CDPET Chips

Code	Heating process					Cooling process	
	$T_g$ (°C)	$T_{ch}$ (°C)	$T_m$ (°C)	$\Delta H_m$ (J/g)	$X_c$ (%)	$T_{cc}$ (°C)	$H_{cc}$ (J/g)
Sample 1	71.2	131.9	253.8	43.8	31.3	193.3	39.8
Sample 2	70.4	142.8	248.6	42.2	30.2	169.5	16.7
Sample 3	69.5	145.1	246.3	40.9	29.2	155.9	14.8
Sample 4	69.2	146.7	243.2	39.3	28.1	154.8	13.6
Sample 5	69.1	149.9	242.1	38.6	27.6	152.6	13.2
Sample 6	68.9	152.8	240.4	36.5	26.1	151.7	12.7
Sample 7	66.3	—	229.5	24.4	17.4	—	—
Sample 8	63.1	—	202.3	6.6	4.7	—	—



**Figure 1** DSC curves of PET, CDPET, and PEG-modified CDPET chips.

melting peak was observed for PET polymer. The higher melting temperatures associated with the melting peak of CDPET and PEG-modified CDPET polymers were also lower than that of PET polymer. Interestingly, large  $-\text{SO}_3\text{Na}$  side groups in CDPET and PEG-modified CDPET polymers prevented the chain molecules from tightly coagulating and obstructed the formation of larger crystals.<sup>16</sup> Meanwhile, the  $T_m$  of the CDPET polymer was higher than that of the PEG-modified CDPET. The effect of the PEG soft segments reduced the  $T_m$  of the PEG-modified CDPET polymer.

The glass-transition temperature ( $T_g$ ), which is related to the initiation of the micro-Brownian motion of the amorphous chains, is between 63 and 72°C for all samples. Clearly, the  $T_g$  values of CDPET and PEG-modified CDPET polymers declined as the SIPM and PEG contents decreased. The PEG soft segments caused the  $T_g$  of the PEG-modified CDPET polymer to be slightly lower than that of the CDPET polymer at the same SIPM content. At fixed SIPM and PEG contents, the  $T_g$  of the PEG2000–CDPET polymer was lower than that of the PEG400–CDPET polymer.

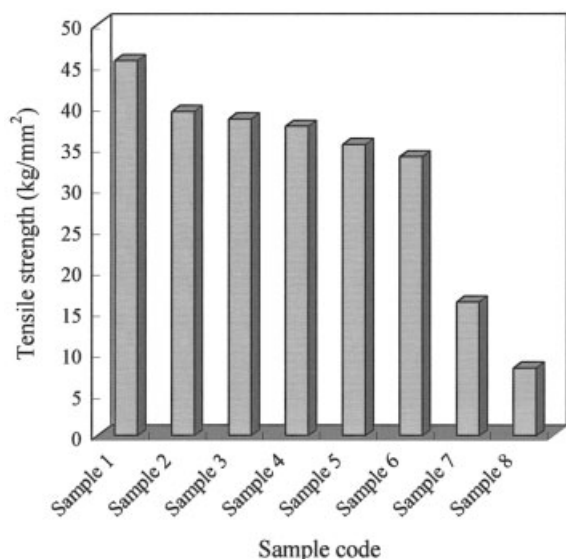
The melting exothermic peaks ( $T_{ch}$ ) of the samples were distributed from 131 to 153°C. Clearly, the  $T_{ch}$  of CDPET and PEG-modified CDPET polymers in-

creased as SIPM and PEG contents increased. The PEG soft segments caused the  $T_{ch}$  of the PEG-modified CDPET polymer to be slightly higher than that of the CDPET polymer with the same SIPM content. The  $T_{ch}$  of the PEG2000–CDPET polymer exceeded that of PEG400–CDPET polymer given the same SIPM and PEG contents.

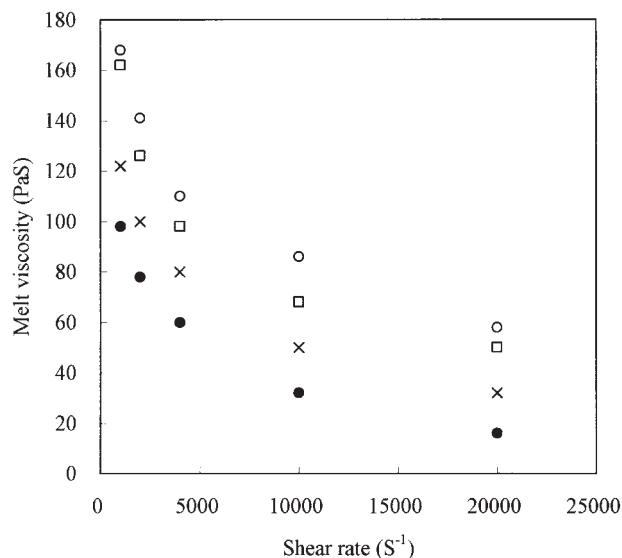
During DSC cooling, the exothermic peak ( $T_{cc}$ ) of the PET polymer associated with crystallization was higher than that of CDPET and PEG-modified CDPET polymers, implying that the rate of crystallization of the PET polymer was greater than that of the CDPET and PEG-modified CDPET polymers. A large  $-\text{SO}_3\text{Na}$  side group in the CDPET polymer inhibits the formation of crystalline particles. Existence of the  $-\text{SO}_3\text{Na}$  side group in the polymer causes the thermal behavior of the PEG-modified CDPET polymer to resemble that of the CDPET polymer. The PEG soft segments cause the  $T_{cc}$  of the PEG-modified CDPET polymer to be lower than that of the CDPET polymer at the same SIPM content. The  $T_{cc}$  of the PEG400–CDPET polymer exceeded that of the PEG2000–CDPET polymer at fixed SIPM and PEG contents.

### Analysis of tensile strength

Figure 2 clearly reveals that the tensile strength of the specimen declined as the SIPM content in the CDPET polymer increased. Analysis of the crystallinity demonstrated that the crystallinity decreased as the SIPM content increased. Hong et al.<sup>17</sup> also reported that the intrinsic viscosity gradually de-



**Figure 2** Tensile strengths of PET, CDPET, and PEG-modified CDPET films.

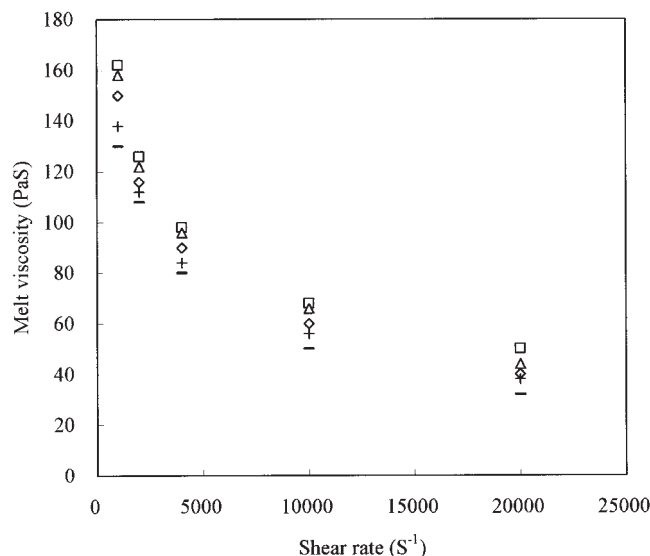


**Figure 3** Melt viscosity of PET and CDPET polymers versus shear rate at 260°C. (○): PET; (□): CDPET-2; (×): CDPET-6; (●): CDPET-10.

clined as the SIPM content increased, indicating a decrease in the molecular weight. Furthermore, adding a compound that contained the  $-\text{SO}_3\text{Na}$  side group disturbed the regularity of the PET polymer chain molecules, rendering the structure of the CDPET polymer less compact than that of the PET polymer, as was reflected in the lower tensile strength of the former polymer. The tensile strengths of the CDPET series polymers followed the order CDPET-2 > CDPET-6 > CDPET-10. The tensile strength of the PEG-modified CDPET polymer slowly diminished as the PEG content increased, at the same SIPM content and a particular  $\bar{M}_n$  of the PEG. At the same SIPM and PEG contents, the tensile strength of the PEG400–CDPET polymer exceeded that of the PEG2000–CDPET polymer. The tensile strengths of the PET, CDPET, and PEG-modified CDPET polymers followed the order PET > CDPET-2 > PEG400-3-CDPET-2 > PEG400-6-CDPET-2 > PEG2000-3-CDPET-2 > PEG2000-6-CDPET-2 > CDPET-6 > CDPET-10.

### Flow behavior

Figure 3 plots the melt viscosities of PET and CDPET polymers at 260°C against shear rate. From 1000 to 20,000 s<sup>-1</sup>, all polymers exhibited pseudoplastic flow. The melt viscosity of the PET polymer exceeded that of the CDPET polymer over the entire range of shear rates. Figure 4 displays the melt viscosities of PEG-modified CDPET materials at 260°C against shear rate. The melt viscosities of PEG-modified CDPET polymers also exhibited pseudoplastic flow. At the same

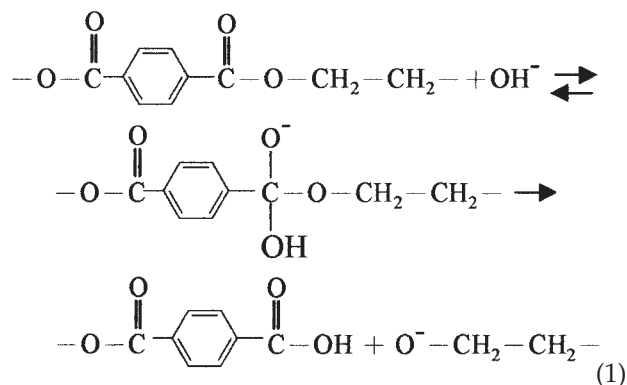


**Figure 4** Melt viscosity of PEG-modified CDPET polymers versus shear rate at 260°C. (□): CDPET-2; (△): PEG400-3-CDPET-2; (◇): PEG400-6-CDPET-2; (+): PEG2000-3-CDPET-2; (-): PEG2000-6-CDPET-2.

SIPM content, the melt viscosity of the PEG-modified CDPET polymer was lower than that of the CDPET polymer. At fixed SIPM and PEG contents, the melt viscosity of the PEG400-CDPET polymer exceeded that of the PEG2000-CDPET polymer.

### Kinetics of alkaline hydrolysis

This work also investigated the kinetics of the alkaline hydrolysis of PET, CDPET, and PEG-modified CDPET materials, using stastical regression analysis. The dissolution of PET can be represented by the following equation<sup>18,19</sup>:



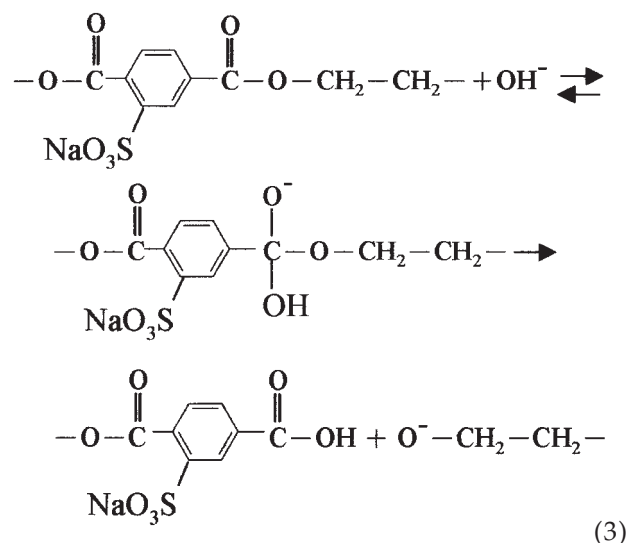
In the first step,  $\text{OH}^-$  in aqueous sodium hydroxide (NaOH) solution attacks  $\begin{array}{c} \text{---C---} \\ \parallel \\ \text{O} \end{array}$  with a lower electron cloud density, producing an intermediate. In the sec-

ond step, a further reaction between  $\text{OH}^-$  and  $\text{---C---}$  breaks the macromolecular chain and produces  $\text{---COOH}$  and  $\text{O}^-\text{---CH}_2\text{---CH}_2\text{---}$ ; the final product is  $\text{---COONa}$  in alkaline solution. Quantitative results imply that the reaction rate is related to the concentration of  $\text{OH}^-$  and  $\begin{array}{c} \text{---C---} \\ \parallel \\ \text{O} \end{array}$  in the reaction system. Therefore, the reaction rate can be expressed as<sup>8</sup>

$$-\frac{dW}{dt} = KC^\alpha A^\beta \quad (2)$$

where  $W$  is the weight of the sample (g);  $t$  is the reaction time (min);  $C$  is the concentration of NaOH (%; g/L);  $A$  is the reactive surface area ( $\text{m}^2$ );  $K$  is the rate constant of alkaline hydrolysis, and  $\alpha$  and  $\beta$  are the reaction progression factors, respectively.

The DSC experimental results prove that the SIPM was not packed into the original polyester unit cell, but concentrated at the polyester crystalline surface and the amorphous domains, because of the introduction of the  $\text{---SO}_3\text{Na}$  functional group and the preference of the  $\text{OH}^-$  group to hydrolyze the CDPET phase over the PET phase. The dissolution of CDPET can be represented by the following equation<sup>20</sup>:



Additionally, the molecular structure of the PEG-modified CDPET polymer closely resembled that of the CDPET polymer. The dissolution of the PEG-modified CDPET can be represented by the following equation, where  $n$  is the degree of polymerization of the PEG molecules.



hydrolysis followed the order CDPET-10 > CDPET-6 > PEG2000-6-CDPET-2 > PEG2000-3-CDPET-2 > PEG400-6-CDPET-2 > PEG400-3-CDPET-2 > CDPET-2 > PET. Notably, the rate constant of the alkaline hydrolysis of CDPET and PEG-modified CDPET polymers increased with SIPM/PEG content and the  $\bar{M}_n$  of the PEG increased.

### CONCLUSIONS

The crystallinities of CDPET and PEG-modified CD-PET materials decreased as the SIPM/PEG content and the  $\bar{M}_n$  of the PEG increased, under fixed process conditions. Clearly, the SIPM and PEG segments did not change the original PET unit cell. The  $-\text{SO}_3\text{Na}$  functional group and the PEG molecules gathered only on the crystal surface and in amorphous regions. The value of  $\beta$  was found to equal 1 from the regression analysis results of kinetics of the alkaline hydrolysis of PET, CDPET, and PEG-modified CDPET materials. Moreover, the  $R^2$  of the kinetic equation for  $\alpha$  from 1.09 to 1.25 was better than that for  $\alpha = 1$ . The results further demonstrate that the rate constants of alkaline hydrolysis followed the order CDPET-10 > CDPET-6 > PEG2000-6-CDPET-2 > PEG2000-3-CDPET-2 > PEG400-6-CDPET-2 > PEG400-3-CDPET-2 > CDPET-2 > PET. Clearly, the rate constants of alkaline hydrolysis of CDPET and PEG-mod-

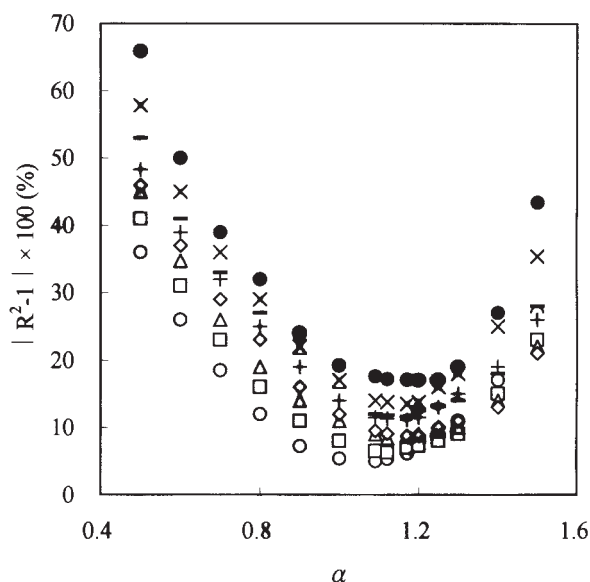
**TABLE IV**  
Kinetics of PET, CDPET, and PEG-Modified CDPET Chips<sup>a</sup>

Code	$K \times 10^{-3}$ (g/min m <sup>-2</sup> %)	$\alpha$	$\beta$	$R^2$
Sample 1	0.043	1	1	0.9964
Sample 1	0.038	1.09	1	0.9990
Sample 2	0.326	1	1	0.9857
Sample 2	0.288	1.12	1	0.9983
Sample 3	0.945	1	1	0.9832
Sample 3	0.835	1.14	1	0.9939
Sample 4	1.847	1	1	0.9884
Sample 4	1.632	1.15	1	0.9962
Sample 5	2.749	1	1	0.9857
Sample 5	2.430	1.16	1	0.9968
Sample 6	3.651	1	1	0.9872
Sample 6	3.227	1.17	1	0.9975
Sample 7	9.064	1	1	0.9853
Sample 7	8.010	1.18	1	0.9949
Sample 8	18.128	1	1	0.9873
Sample 8	16.020	1.25	1	0.9943

<sup>a</sup> Surface area of 5-g chips was approximately 0.007 m<sup>2</sup>.

ified CDPET polymers increased with the SIPM/PEG content and the  $\bar{M}_n$  of the PEG increased.

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**Figure 7** Relationship between  $|R^2 - 1|$  and  $\alpha$ , when  $\beta$  equals 1. (○): PET; (□): CDPET-2; (△): PEG400-3-CDPET-2; (◇): PEG400-6-CDPET-2; (+): PEG2000-3-CDPET-2; (-): PEG2000-6-CDPET-2; (×): CDPET-6; (●): CDPET-10.

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