

Study of Glycolysis of Poly(ethylene terephthalate) Recycled from Postconsumer Soft-Drink Bottles. III. Further Investigation

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ABSTRACT: A modified glycolysis reaction of recycled poly(ethylene terephthalate) (PET) bottles by ethylene glycol (EG) was investigated. Influences of the glycolysis temperature, the glycolysis time, and the amount of catalysts (per kg of recycled PET) were illustrated in this study. The manganese acetate was used as a glycolysis catalyst in this study. Bis-2-hydroxyethyl terephthalate (BHET) and its dimer were predominately glycolysis products. It was found the optimum glycolysis temperature is 190°C. And the best glycolysis condition is 190°C of glycolysis temperature, 1.5 h of glycolysis time, and 0.025 moles of manganese acetate based on per kg of recycled PET. If the best glycolysis condition is conducted, the glycolysis conversion may be as high as 100%. For a given reaction time (1.0 h), the $\ln(\%$ glycolysis conversion) is linear to $1/T$ (K^{-1}) and the activation energy (E) of glycolysis reaction is around 92.175 kJ/(g mole). The glycolysis conversion rate increases significantly with increasing the glycolysis temperature, the glycolysis time, or

the amount of manganese acetate (glycolysis catalyst). Thermal analyses of glycolysis products were examined by a differential scanning calorimetry (DSC) and a thermogravimetric analysis (TGA). According to the definition of a 2^3 factorial experimental design, the sequence of the main effects on the glycolysis conversion of the recycled PET, in ascending order, is the glycolysis time (0.18) < the amount of catalyst per kg of the recycled PET (0.34) < the glycolysis temperature (0.40). Meanwhile, the prediction equation of glycolysis conversion from the result of a 2^3 factorial experimental design is $\hat{Y} = 0.259 + 0.20X_1 + 0.09X_2 + 0.17X_3 + 0.06X_1X_2 + 0.145X_1X_3 + 0.05X_2X_3 + 0.035X_1X_2X_3$. © 2003 Wiley Periodicals, Inc. *J Appl Polym Sci* 87: 2004–2010, 2003

Key words: glycolysis; ethylene glycol; recycled poly(ethylene terephthalate); manganese acetate; factorial experimental design

INTRODUCTION

Poly(ethylene terephthalate) (PET) is a thermoplastic polyester. Due to its characteristic of high strength, transparency, and safety, PET is used widely for many applications. Currently, PET in the amorphous state is applied a good deal in the manufacturing of soft-drink bottles for replacing poly(vinyl chloride) (PVC) and glass bottles. Since PET bottles do not decompose in the nature readily, the disposal of a large amount of PET bottles has caused serious environmental problems.^{1,2} After the green revolution movement started in the 1980s, researchers have begun to focus on the recycling and management of plastics waste.^{3,4} Today, PET bottles have become one of the most valuable and successfully recyclable materials.

There are various possibilities for dealing with recycled PET bottles. These PET recycled methods include (1) hydrolysis with acids or bases in the aqueous solution,^{5,6} (2) noncatalytic hydrolysis or alcoholysis in the supercritical media,^{7,8} or (3) alcoholysis or gly-

colysis by catalytic reactions.^{9–11} Since glycolysis of recycled PET bottles can produce the bis-2-hydroxyethyl terephthalate (BHET) monomer and BHET has been widely used in the production of unsaturated polyesters and rigid or flexible polyurethanes,⁹ the glycolysis process is chosen for further investigation in this study.

Kamal et al.⁷ studied the reaction of PET melts with ethylene glycol (EG) in a pressurized reactor at the temperature above 245°C. They concluded that the reaction rate was dependent on temperature, concentration of EG, and ethylene diester group in the polymer. Chen et al.⁸ also had illustrated the kinetics of the glycolysis depolymerization of PET in a pressurized reactor at temperatures between 190 and 240°C. They found that PET depolymerization was dependent on pressure, temperature, and EG/PET ratio. They also demonstrated that the glycolysis rate was the second order with respect to the EG concentration. Meanwhile, EG acting as reactant and catalyst in the glycolysis reaction was mentioned in their report. Baliga and Wong¹¹ had conducted glycolysis experiments of the PET recycled from postconsumer soft-drink bottles. In their study, the glycolysis temperature was brought up to 190°C and the glycolysis reaction was

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allowed to proceed up to 8 h (with small samples removed periodically to monitor the progress of the glycolysis reaction). They found that zinc acetate was the best catalyst in terms of the extent of glycolysis of four metal acetates (lead, zinc, cobalt, and manganese) tested, yielding the highest amount of BHET monomer formed. Instead of using molar ratio, the amount of metal acetate used in their study was 0.48 g, which was equal to 0.5% by the weight of PET.

If the amount of metal acetate was in moles per kg of the recycled PET instead of in weights, it was found that the best glycolysis catalyst was manganese acetate.¹² Therefore, in this research, the various moles of manganese acetate per kg of the recycled PET were used. The influences of glycolysis conditions (temperature, time, and the amount of catalyst) on the glycolysis of the recycled PET were systemically studied. In addition, the glycolysis product labeled as the X fraction in the previous work¹³ was divided into two parts in this study. One part was labeled as the X1 fraction. The composition of the X1 fraction was the same as the X fraction. On the other hand, the other part was put into 100°C boiling water and then filtered. The filter cake was dried and labeled as the X2 fraction. And, the filtrate was collected and stored in a refrigerator for 24 h. The white crystalline flake of BHET was formed in the filtrate. In order to compare with the previous work,¹³ the thermal properties of glycolysis products were also discussed here.

Also, in order to realize the main interaction, two-factor interaction, and three-factor interaction effects of these three independent glycolysis variables on the glycolysis conversion of the recycled PET, a 2³ factorial experimental design [three independent variables with high (+), and low (-) levels] was conducted and a prediction equation of glycolysis conversion from the result of a 2³ factorial experimental design was also illustrated here. In addition, in order to compare with the previous work,¹⁴ the low level of the amount of catalyst chosen was 0.0025 moles per kg of the recycled PET instead of 0.00 moles—because without any catalyst, there is no significant amount of the glycolysis product and no equation that interpolates between those levels is meaningful. From the results of this 2³ factorial experimental design, the impact of the levels of the independent glycolysis variables on the main the two-factor interaction and the three-factor interaction effects can be illustrated.

EXPERIMENTAL

Materials

Commercially clear PET soft-drink bottles were recycled, washed, dried, and cut into 0.4 × 0.4 cm flakes for glycolysis experiments. These recycled PET bottles were blow-molding grade (IV = 0.85 in 60% phenol/

40% tetrachloroethane). Both EG and manganese acetate were purchased from Katayama Chemical. All reagents were used without further purification or treatment.

Glycolysis Experiment

A 1000-mL, four-necked, flat-bottom reactor was used for all glycolysis experiments. This reactor was equipped with a thermometer and a reflux condenser. A magnetic stirrer was put in the reactor to ensure proper mixing. The EG/PET ratio used in the glycolysis experiments was 2:1 (w/w). The weights of EG and the recycled PET were 160 and 80 g, respectively. Therefore, the total weight of recycled PETs was 240 g. In order to ensure limited water content in the reactor, the reactor was heated up to 110°C and held at this temperature for at least 2 min.

In order to realize the influences of the glycolysis temperature, the glycolysis time, and the catalyst concentration on the depolymerization of the recycled PET, three groups of glycolysis experiments were designed as below:

- I. The glycolysis temperature was set at 190°C and 0.025 moles of manganese acetate per kg of the recycled PET was used. The glycolysis time was varied from 0 to 2 h with 0.5 h interval.
- II. The glycolysis temperature was 190°C and the glycolysis time was 1.0 h. The molar amount of manganese acetate was varied from 0 to 0.075 moles per kg of the recycled PET.
- III. The glycolysis time was 1.0 hr and 0.025 moles of manganese acetate per kg of the recycled PET were used. The glycolysis temperature was varied from 110 to 190°C.

After each specified glycolysis experiment was completed, the reactor was removed from the hot plate and 200 mL boiling water was added into the reactor slowly. Then, a copper screen with 0.05 × 0.05 cm pore size was used to filter the material quickly. The undepolymerized recycled PET was collected, dried, weighed, and labeled as the PET fraction. The conversion percentage for the glycolysis of the recycled PET was defined as eq. (1):

Glycolysis conversion percentage

$$= \frac{A - B}{A} \times 100\% \quad (1)$$

where *A* represents 80 g recycled PET and *B* represents the weight of undepolymerized recycled PET (PET fraction). Meanwhile, the residual glycolysis product mixture was filtered. The filtrate was collected and stored in a refrigerator for 24 h. The white

crystalline flakes of BHET were formed in the filtrate and then filtered. The white crystalline flakes of BHET was washed by the 600 mL cooled distilled water in order to remove the manganese acetate. The white crystalline flakes of BHET was collected, dried, and labeled as the BHET fraction.

The filter cake was collected and divided into two parts. One part was dried and labeled as the X1 fraction. In order to remove the residual BHET from the filter cake, the other part was put into 600 mL 100°C boiling water and filtered. The filter cake was dried and labeled as the X2 fraction. Then, the filtrate was collected and stored in a refrigerator for 24 h. The white crystalline flakes of BHET were formed in the filtrate.

Characterization

The hydroxyl values of BHET, X1, and X2 fractions were all analyzed according to the ASTM D2849 Method A. A differential scanning calorimeter (DSC) (Perkin Elmer Model DSC 7) was also used for the thermal analyses of the BHET, the X1, and the X2 fractions. These samples were heated from room temperature to a preset temperature with a heating rate of 20°C/min (under a nitrogen stream).

A thermogravimeter analyzer (TGA) (Perkin Elmer Model TGA 7) was used to measure the weight loss of BHET fraction, X1 fraction, and X2 fraction under a nitrogen stream during a temperature scan from 50 to 600°C with a heating rate of 20°C/min. The chemical structure of glycolysis products examined by a Fourier-transform infrared spectrophotometer (FTIR) had been clearly illustrated in the previous work.¹³

Experimental Design

The glycolysis temperature (X_1), the glycolysis time (X_2), and the amount of catalyst (X_3) were chosen as the independent variables. Two levels, high (+) and low (-), were also defined for each independent variable. Thus, the 2³ factorial experimental design had eight runs, the first in standard order being (- - -), and the last in standard order being (+ + +). The gly-

TABLE I
Influence of the Glycolysis Time on the Glycolysis Conversions of the Recycled PET

Glycolysis time (h)	Glycolysis conversion (%)
0	0.00
0.5	54.20
1.0	98.48
1.5	99.47
2.0	99.96
2.5	100.00

Glycolysis temperature = 190°C and amount of manganese acetate = 0.025 moles per kg of the recycled PET.

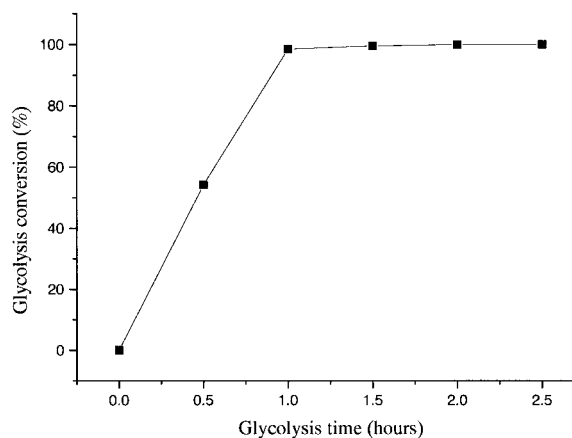


Figure 1 The relationship between the glycolysis conversion of the recycled PET and the glycolysis time (190°C glycolysis temperature and 0.025 moles of manganese acetate per kg of the recycled PET).

colysis conversion of the recycled PET was studied as the dependent variable here. For glycolysis temperature, 190 and 130°C were chosen as high and low levels, respectively; 2.0 and 0.5 h were chosen as high and low levels for glycolysis time, respectively. For the amount of catalyst, 0.025 and 0.0025 moles per kg of the recycled PET were chosen as high and low levels, respectively. Here, the low level of the amount of catalyst was 0.0025 moles instead of 0.00 mole, since without any catalyst, there is no significant amount of the glycolysis product and no equation that interpolates between those levels is meaningful.

RESULTS AND DISCUSSION

The result of elemental analysis of BHET fraction was C = 56.73%, H = 5.41%, and O = 37.86%. This result was similar to theoretical result of BHET (C = 56.74%, H = 5.5%, and O = 37.76%). The oxygen content in the elemental analysis result was a little bit higher than that in the theoretical result. This might be due to the presence of moisture. The theoretical hydroxyl values of pure BHET monomer and its dimer were 441.5 and 251.3 mg KOH/g, respectively.¹¹ In this study, the hydroxyl values of the BHET fraction, the X1 fraction, and the X2 fraction were analyzed according to ASTM D2849 Method A. The hydroxyl numbers of the BHET fraction, the X1 fraction, and the X2 fraction were 438.0, 338.4, and 252.2 mg KOH/g, respectively. The hydroxyl value of the X1 fraction was lower than that of the BHET but higher than that of the dimer. This implies that the X1 fraction was a mixture of BHET and dimer. Besides, the hydroxyl value of the X2 fraction was similar to that of dimer of BHET. This indicates that X2 fraction was almost a dimer of BHET.

The relationship between the glycolysis conversion of the recycled PET and the glycolysis time is shown in Table I and Figure 1. The glycolysis temperature was

TABLE II
Influence of the Amount of Manganese Acetate on the Glycolysis Conversions of the Recycled PET

Moles of catalyst per kg of the recycled PET	Glycolysis conversion (%)
0	0.00
0.0025	15.21
0.0125	91.78
0.025	98.48
0.0375	99.99
0.050	100.00
0.0625	100.00
0.075	100.00

Glycolysis temperature = 190°C and glycolysis time = 1.0 h.

set at 190°C, and 0.025 moles of manganese acetate per kg of the recycled PET were used. Generally speaking, the glycolysis conversion increases with increasing the glycolysis time. The glycolysis conversion will eventually approach a steady value when the glycolysis time is longer than 1.5 h. In Figure 1, the glycolysis conversion is 54.2% at 0.5 h glycolysis time. And the glycolysis conversion is almost proportional to the first order of glycolysis time from 0.5 to 1.0 h. However, if the glycolysis time is extended to 1.0 h, the glycolysis conversion increases dramatically up to 98%. When the glycolysis time is extended to 1.5 h or longer, the glycolysis conversion is almost 100%.

Table II and Figure 2 indicate the relationship between the glycolysis conversion of the recycled PET and the amount of manganese acetate. This experiment condition was set as 190°C glycolysis temperature and 1.0 h glycolysis time. In Figure 2, the glycolysis conversion increases with increasing the amount of manganese acetate per kg of the recycled PET. And the conversion can eventually approach a steady conversion when the amount of manganese acetate is more than 0.025 moles based on per kg of the recycled

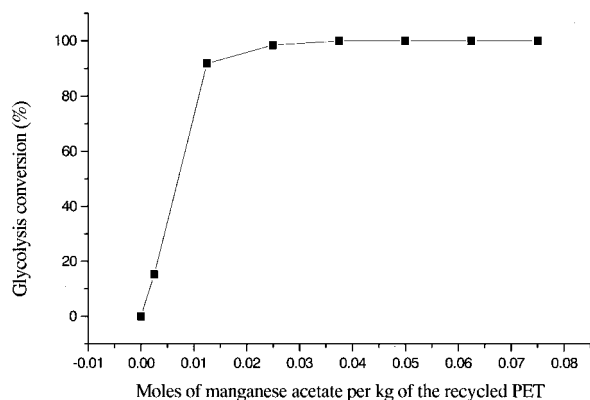


Figure 2 The relationship between the glycolysis conversion of the recycled PET and the amount of manganese acetate per kg of recycled PET (190°C glycolysis temperature and 1.0 h glycolysis time).

TABLE III
Influence of Glycolysis Temperature on the Glycolysis Conversions of the Recycled PET

Glycolysis temperature (°C)	Glycolysis conversion (%)
110	0
130	3.12
140	5.86
150	8.92
160	17.86
170	35.16
180	68.46
190	98.48

Glycolysis time = 1.0 h and amount of manganese acetate = 0.025 moles per kg of the recycled PET.

PET. Figure 2 also illustrates the glycolysis conversion is about 91% if 0.0125 moles of manganese acetate (per kg of the recycled PET) is used and the glycolysis conversion is almost proportional to the first order of amount of manganese acetate. Moreover, if 0.025 moles manganese acetate (per kg of recycled PET) are used, the glycolysis conversion increases up to 98%. If the amount of manganese acetate is higher than 0.025 moles per kg of recycled PET, the glycolysis conversion is almost a constant (approximately 100%).

Table III and Figure 3 show the relationship between the glycolysis conversion of the recycled PET and the glycolysis temperature. The figure is generated under the situation that glycolysis time was 1.0 h and 0.025 moles manganese acetate per kg of the recycled PET were used. It shows that the glycolysis conversion is very close to 0% if the glycolysis temperature is below 130°C. When the glycolysis temperature is higher than 150°C, the glycolysis conversion increases with increasing the glycolysis temperature apparently. When the glycolysis temperature is 170°C, the glycolysis conversion reaches 35%. Eventually, the glycolysis conversion increases dramatically from 35

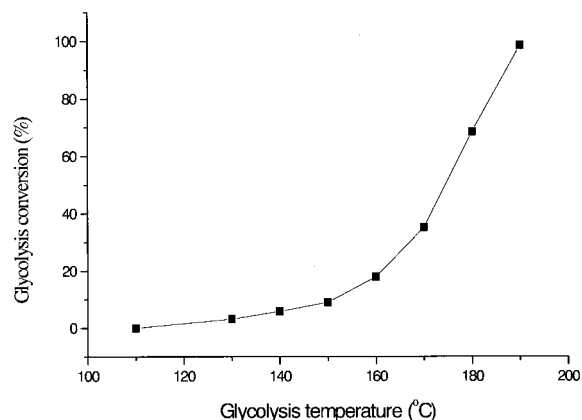


Figure 3 The relationship between the glycolysis conversion of the recycled PET and the glycolysis temperature (1.0 h glycolysis time and 0.025 moles of manganese acetate per kg of the recycled PET).

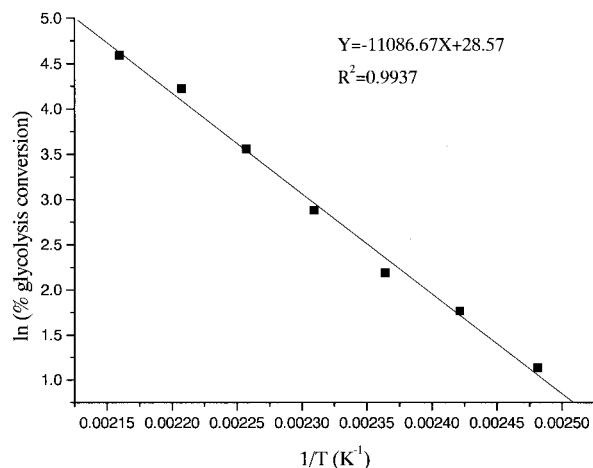


Figure 4 The relationship between $\ln(\% \text{ glycolysis conversion})$ and $1/T$ (1.0 h glycolysis time and 0.025 moles manganese acetate per kg of the recycled PET).

up to 98% when the glycolysis temperature is 190°C. Therefore, the glycolysis temperature is a very critical factor for the glycolysis conversion of the recycled PET.

Since percent glycolysis conversion for a given reaction time (1.0 h) is effectively reaction rate and also proportional to the rate constant (k). Therefore, we can use the data from Table III and plot $\ln(\% \text{ glycolysis conversion})$ vs $1/T$ (K^{-1}). This result is shown in Figure 4. The $\ln(\% \text{ glycolysis conversion})$ is linear to $1/T$ (K^{-1}). The regression equation for this linearity is

$$\ln(\% \text{ glycolysis conversion}) = -11086.67(1/T) + 28.57 \dots (2)$$

$$R^2 = 0.9937$$

Moreover, the slope of $\ln(\% \text{ glycolysis conversion})$ vs $1/T$ (K^{-1}) is equal to that of $\ln(k)$ vs $1/T$ (K^{-1}) since % glycolysis conversion is proportional to the rate constant (k). However, the intercepts of $\ln(\% \text{ glycolysis conversion})$ vs $1/T$ (K^{-1}) and $\ln(k)$ vs $1/T$ (K^{-1}) are not the same, unless $\ln(\% \text{ glycolysis conversion})$ is equal to $\ln(k)$. From eq. (2), we can estimate that the activation energy from the slope is -11086.67 . Since the slope (-11086.67) is equal to $-E/R$ and R is equal to $8.314 \text{ J}/(\text{g mole})(\text{K})$, the activation energy (E) is around $92.175 \text{ kJ}/(\text{g mole})$ for a given reaction time (1.0 h).

There are three DSC thermal analysis curves in Figure 5. The DSC thermal analysis of the BHET fraction is illustrated as curve a. The melting onset temperature and the peak temperature of BHET fraction shown in curve a are 107 and 110°C, respectively. Curve b displays the DSC thermal analysis of the X1 fraction. There are two endothermic peaks in curve b. One peak around 105°C can be reasonable associated

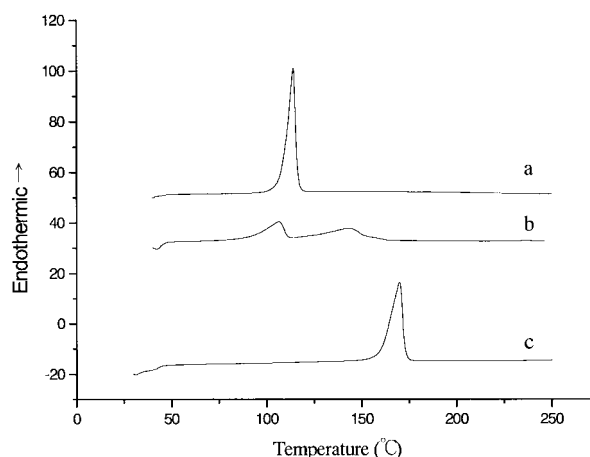


Figure 5 DSC thermal analysis curves of (a) the BHET fraction, (b) the X1 fraction, and (c) the X2 fraction (under nitrogen atmosphere, heating rate = 20°C/min).

with the melting temperature of BHET, whereas the other peak, centered at around 143°C, is quite broad and hence is not readily considered as the melting of dimer. According to Lin's⁸ and Baliga's¹¹ reports, the known melting temperatures of BHET and dimer are around 110 and 170°C, respectively. Therefore, the X1 fraction can be assumed to contain mainly BHET and its dimer. Based upon the research result, it seems to suggest that there are some interactions between the monomer and the dimer at the elevated temperature from this DSC thermal analysis.¹¹ This well agrees with the results of Baliga¹¹ and Tomita.¹⁵ Curve c displays the DSC thermal analysis of the X2 fraction. Apparently, the endothermic peak, centered at around 172°C, is considered as the melting point of dimer.^{11,15}

There are three TGA curves in Figure 6. These curves all indicate a weight loss starting around 430 ~ 450°C. Curve a shows the TGA curve of the recycled PET without the glycolysis reaction. Since the only one

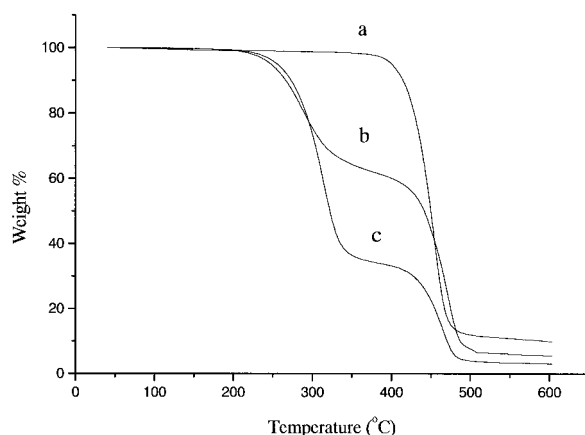


Figure 6 TGA curves of (a) the recycled PET, (b) the X2 fraction, and (c) the BHET fraction (under nitrogen atmosphere, heating rate = 20°C/min).

and major weight loss in the curve a starts around 430 ~ 450°C, the thermal degradation temperature of the recycled PET without the glycolysis reaction is around 430 ~ 450°C. Curve b displays the TGA curve of the X2 fraction. The first weight loss is about 35% and starts around 250 ~ 270°C. This phenomena is attributed to the thermal degradation of the X2 fraction. The second weight loss of about 55% starting around 430 ~ 450°C is attributed to the thermal degradation of the PET produced by the thermal polymerization of the dimer of BHET during the thermogravimetric analysis process. Curve c displays the TGA curve of the BHET fraction. It also shows two major weight loss processes. The first weight loss is about 65% and also starts around 250 ~ 270°C. This phenomena is attributed to the thermal degradation of the BHET fraction. The second weight loss of about 30% begins around 430 ~ 450°C. This weight loss is also due to the thermal degradation of the PET produced by the thermal polymerization of BHET during the thermogravimetric analysis process. This result implies that the weight of the PET produced by the thermal polymerization of BHET dimer is higher than that of BHET during the thermogravimetric analysis process.

Figure 7 shows the relationship between the observed yields (glycolysis conversion) and the standard ordering of a 2³ factorial experimental design of glycolysis experiments for the recycled PET. According to the calculation definition of a 2³ factorial experimental design, the main effects of the glycolysis temperature, the glycolysis time, and the amount of catalyst, are 0.40, 0.18, and 0.34, respectively. Similarly, according to the definition, the two-factor interaction effects of the glycolysis temperature vs the glycolysis time (X₁ vs X₂), the glycolysis temperature vs the amount of catalyst per kg of the recycled PET (X₁ vs X₃), and the

TABLE IV
Summary of the Main Interaction, the Two-Factor Interaction, and the Three-Factor Interaction Effects of the Glycolysis Conversion of the Recycled PET

Main effect	Two-factor interaction effect	Three-factor interaction effect
X ₁ = 0.40	X ₁ vs X ₂ = 0.12	X ₁ vs X ₂ vs X ₃ = 0.07
X ₂ = 0.18	X ₁ vs X ₃ = 0.29	
X ₃ = 0.34	X ₂ vs X ₃ = 0.10	

glycolysis time vs the amount of catalyst (X₂ vs X₃), are 0.12, 0.29, and 0.10, respectively. The three-factor interaction effect of the glycolysis temperature vs the glycolysis time vs. the amount of catalyst per kg of the recycled PET (X₁ vs X₂ vs X₃) is 0.07.

Table IV illustrates the summary of the main interaction, two-factor interaction, and three-factor interaction effects of the glycolysis conversion of the recycled PET. It shows that the sequence of the main effects on the glycolysis conversion of the recycled PET bottles in ascending order is the glycolysis time (0.18) < the amount of catalyst per kg of the recycled PET (0.34) < the glycolysis temperature (0.40). This is because the glycolysis of the recycled PET cannot occur easily without enough thermal energy, even under the condition of high levels of the amount of catalyst and the glycolysis time. Similarly, the amount of catalyst affects the glycolysis conversion of the recycled PET more significantly than glycolysis time does under the same glycolysis temperature condition. Therefore, the glycolysis temperature and the amount of catalyst per kg of the recycled PET represent the first and the second important factors, respectively.

The sequence of the two-factor interaction effects on the glycolysis conversion of the recycled PET, in ascending order, is the glycolysis time vs the amount of catalyst per kg of the recycled PET (0.10) < the glycolysis temperature vs the glycolysis time (0.12) < the glycolysis temperature vs the amount of catalyst per kg of the recycled PET (0.29). As mentioned above, the glycolysis temperature is the most important individual factor. Furthermore, the amount of catalyst per kg of the recycled PET is the second important individual factor. Therefore, the interaction effect between the glycolysis temperature and the amount of catalyst per kg of the recycled PET is the highest in determining the glycolysis conversion of the recycled PET. Similarly, the interaction effect between the glycolysis temperature and the glycolysis time is the second in order. In addition, the interaction effect between the amount of catalyst per kg of the recycled PET and the glycolysis time is the third in order. The three-factor interaction effect, 0.07, is still significantly related to the glycolysis conversion of the recycled PET. From the results of Table IV, we can obtain a prediction equation¹⁶ by the definition of a 2³ factorial experi-

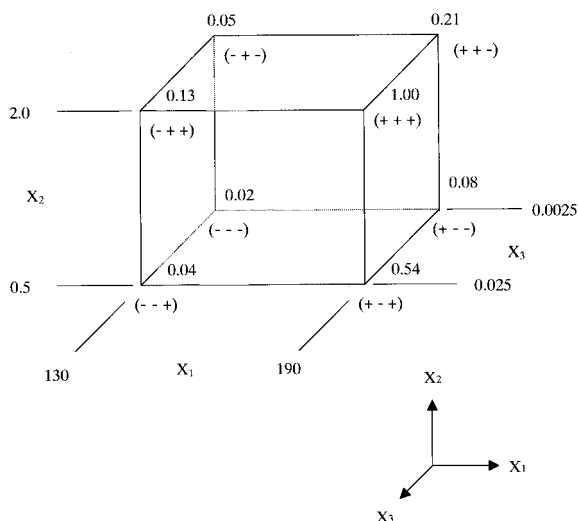


Figure 7 The diagrammatic representation of the observed yields (the glycolysis conversions) and the 2³ standard ordering of glycolysis experiments of the recycled PET.

mental design: $\hat{Y} = Y + 0.20X_1 + 0.09X_2 + 0.17X_3 + 0.06X_1X_2 + 0.145X_1X_3 + 0.05X_2X_3 + 0.035X_1X_2X_3$, where \hat{Y} is the predicted response, $Y = 0.259$ (the average of all response values from the experimental data), and $X_1, X_2, X_3 = +1$ (if high level) or -1 (if low level).

CONCLUSIONS

Influences of the glycolysis temperature, the glycolysis time, and the amount of catalyst (manganese acetate) per kg of the recycled PET have been illustrated. The glycolysis conversion rate increases significantly with the glycolysis temperature, the glycolysis time, and the amount of manganese acetate per kg of the recycled PET. Nearly no glycolysis conversion occurred without adding catalyst or at the glycolysis temperature below 130°C. For a given reaction time (1.0 h), the plot of $\ln(\% \text{ glycolysis conversion})$ vs $1/T$ (K^{-1}) is linear. The activation energy (E) is around 92.175 kJ/(g mole). The result of DSC indicates that BHET and its dimer are predominately glycolysis products. The TGA curves of both the BHET and the X2 fractions show two major weight loss processes. The second weight loss starting around 430 ~ 450°C is attributed to the thermal degradation of the PET produced by the thermal polymerization of monomers or dimers during the thermogravimetric analysis process.

We successfully applied a 2^3 factorial experimental design to study the main interaction, two-factor interaction, and three-factor interaction effects of the glycolysis temperature, the glycolysis time, and the catalyst amount per kg of recycled PET on the glycolysis conversion of the recycled PET. The sequence of the main effects on the glycolysis conversion of the recycled PET, in ascending order, is the glycolysis time

(0.18) < the amount of catalyst per kg of recycled PET (0.34) < the glycolysis temperature (0.40). Also, the prediction equation of the glycolysis conversion from the result of a 2^3 factorial experimental design is $\hat{Y} = 0.259 + 0.20X_1 + 0.09X_2 + 0.17X_3 + 0.06X_1X_2 + 0.145X_1X_3 + 0.05X_2X_3 + 0.035X_1X_2X_3$.

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