

Original Research Article**Alkaline Solvolysis of Poly(ethylene terephthalate) in Butan-1-ol Media: Kinetics and Optimization studies****ABSTRACT**

This study was carried out to examine the thermo-chemical decomposition of postconsumer poly(ethylene terephthalate) (PET) in alkaline solution of butan-1-ol. The effect of various process parameters such as reactor temperature, time and sodium hydroxide concentration on the degree of PET degradation and products yield were studied and it was found that the decomposition of PET was essentially complete in an hour, with terephthalic acid and ethylene glycol being the main products. A kinetic study of the process showed that the alkaline solvolysis of PET is a second order reaction.

1. Introduction

Poly(ethylene terephthalate) (PET) is a linear thermoplastic, bluish-white resin made from terephthalic acid and ethylene glycol through poly-condensation (Aguado *et al.*, 1999). It is an indispensable material owing to its low cost (Thompson *et al.*, 2009), excellent tensile strength, chemical resistance, clarity, processability, and reasonable thermal stability (Caldicott, 1999). It is mainly applied in the textile industry, where more than 60% of the entire PET produced worldwide is consumed. Enormous amounts are also used for other applications including manufacture of video and audio tapes, X-ray films, thermoformed products and food packaging (Carragher, 2000; ILSI, 2000; Olabisi, 1997). In food packaging, PET has become the choice especially for beverages mainly due to its glass-like transparency coupled with adequate gas barrier properties for retention of carbonation. It provides an excellent barrier against oxygen and carbon dioxide in the carbonated soft drink sector, which has been growing more rapidly than other applications. In addition, it exhibits a high toughness/weight property ratio, which allows lightweight and securely unbreakable containers with large capacity (Welle, 2011).

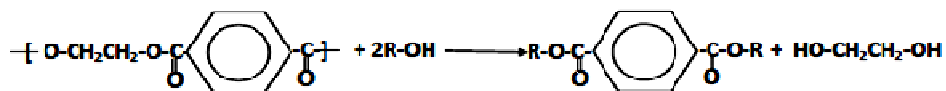
Combining the relative simplicity of the polymerization process with the durable mechanical properties of PET, industries throughout the world have used PET as the staple polymer for beverage packaging. Along with this widespread use of PET is the inevitable creation of large amounts of post-consumer PET waste due to its increasing consumption rate and non-biodegradability, thus creating serious environmental concerns. PET does not have any side effects on the human body, and does not create a direct hazard to the environment. However, due to its substantial fraction by volume in the waste stream and its high resistance to the atmospheric and biological agents, it is considered as a noxious material (Paszun and

32 Spychaj, 1997). With the increase in the amount of PET wastes, its disposal began to pose serious
 33 economical and environmental problems. The recycling of PET does not only serve as a partial solution to
 34 the solid waste problem but also contributes to the conservation of raw petrochemical products and
 35 energy. Products made from recycled plastics can result in 50-60% capital saving as compared to making
 36 the same product from virgin resin (Sinha *et al.*, 2008).

37 Recycling is assumed to be one of the best approaches to solve the problems of PET wasted
 38 accumulation. Various methods such as primary recycling (Hopewell *et al.*, 2009; Al-Salem *et al.*, 2009),
 39 mechanical recycling (Aguado and Serrano, 1999; Bartolome *et al.*, 2012) and chemical recycling
 40 (Campanelli *et al.*, 1993; Yoshioka *et al.*, 2003; Hopewell *et al.*, 2009; IPTS 2012) have been considered
 41 to recycle the PET. Among the various methods of PET recycling, only chemical recycling conforms to
 42 the principles of sustainable development because it has potentials for yielding the raw materials from
 43 which PET is originally made. However, the various chemical recycling routes reported in various works
 44 (such as hydrolysis, alcoholysis and aminolysis) are fraught with shortcomings such as the need for high
 45 pressure and corrosion resistant equipment (Genta *et al.*, 2007), high temperature (Campanelli *et al.*,
 46 1993), and long reaction times (Yoshioka *et al.*, 2003). Taking into account the cost and energy
 47 consumption, chemical reclamation methods are the most effective recycling methods for PET. Alkaline
 48 solvolysis was chosen for this study because it operates under less hazardous conditions, effectively
 49 eliminating the need for corrosion resistant pressure vessels (since it can be carried out at atmospheric
 50 pressure).

51 Preliminary studies on the alkaline solvolysis of postconsumer PET in methanol or ethanol media showed
 52 that it is possible to decompose PET according to the reaction mechanisms shown below (Sanda *et al.*,
 53 2012):

54 **Step 1:** The long polyester chain in PET is broken down using a suitable alcohol. This yields the
 55 corresponding dialkyl terephthalate and ethylene glycol:



56 This step is the alcoholysis step, similar to the methanolysis process reported by Kondo and Genta (2001)
 57

58 **Step 2:** The dialkyl terephthalate from the alcoholysis step reacts with an alkali such as sodium hydroxide
 59 (NaOH) to yield the corresponding salt:



60

2

61 **Step 3:** The sodium salt can be broken down to yield terephthalic acid and the corresponding salt of the
 62 alkali used using a mineral acid:



63
 64 where HX is a monobasic acid such as HCl or HNO₃.

65 The aim of this study is to explore the applicability of a higher molecular weight primary alcohol such as
 66 butan-1-ol for the alkaline solvolysis of PET with a view to examining the effects of various parameters
 67 such as process temperature, reaction time and alkali dosage on the process. The study also examined the
 68 various products formed in the decomposition process and proposes reaction mechanisms where possible.

69 2. Experimental

70 2.1. Materials

71 Postconsumer PET bottles used for the study were sourced from restaurants, fast foods outlets and hotels
 72 in Ile-Ife, Nigeria. Butan-1-ol (Lobachemie), sodium hydroxide (J. T. Baker) and pyridine (Merck
 73 Millipore) were obtained from reputable chemical stores in Lagos, Nigeria. All reagents were used as-
 74 received.

75 The PET bottles collected were shredded after separating from the non-PET components such as labels
 76 and caps. The shredded PET pieces were washed and dried in an oven at 110 °C for 4 h and after drying,
 77 stored in airtight plastic containers prior use.

78 2.2 Alkaline Solvolysis of PET

79 For each run, about 5 g of PET flakes was placed in a two-neck 500 cm³ flask with 100 cm³ of 40 g/L
 80 sodium hydroxide in butan-1-ol. The flask was dipped in a thermostatic oil bath with the temperature of
 81 the bath maintained between 120 and 150 °C. The mixture was heated for 10 – 60 min under reflux and at
 82 the end of each run, 100 ml of distilled water was added to dissolve the solid products. The mixture was
 83 then filtered through a Whatman No. 1 filter paper to separate the unreacted PET from the mother liquor
 84 and the residue was washed with additional 50 cm³ of distilled water. Terephthalic acid (TPA) was
 85 precipitated from the filtrate by the addition of 1 M hydrochloric acid and removed with a Whatman No.
 86 1 filter paper. The unreacted PET and the TPA produced were dried in an oven for 2 h at 105 °C and
 87 weighed. The percent decomposition of PET was determined by gravimetry using the expression below:

$$88 \quad \% \text{ Decomposition of PET} = \left(\frac{W_o - W_f}{W_o} \right) \times 100 \quad (1)$$

89 where W_o is the initial mass of PET flakes and W_f represents the mass of unreacted PET at the end of
 90 experiment. The terephthalic acid yield is given by:

$$91 \quad \text{TPA yield} = \frac{\text{mass of terephthalic acid produced}}{\text{mass of PET fed into the reactor}} \quad (2)$$

92 In order to optimize the alkaline solvolysis process, a three-level-three factor Box-Behnken design was
 93 employed for this study, with 15 experimental runs per alcohol. The factors investigated in this study
 94 were temperature ($^{\circ}\text{C}$), reaction time (min) and alkali concentration (g/l). The coded and uncoded levels
 95 of the independent factors are shown in Table 1.

96 The response for each alcohol was evaluated using Minitab statistical software (version 16.1.1) and fitted
 97 to the quadratic model below:

$$98 \quad Y = \delta_o + \delta_1 X_1 + \delta_2 X_2 + \delta_3 X_3 + \delta_{12} X_1 X_2 + \delta_{13} X_1 X_3 + \delta_{23} X_2 X_3 + \delta_{11} X_1^2 + \delta_{22} X_2^2 + \delta_{33} X_3^2 \quad (3)$$

99 Where Y is the predicted response (% PET decomposition or TPA yield), δ_o is the intercept term, δ_1 , δ_2 ,
 100 δ_3 are the linear coefficients, δ_{12} , δ_{13} , δ_{23} are the interactive coefficients and δ_{11} , δ_{22} , δ_{33} are the
 101 quadratic coefficients. In addition, the terms X_1 , X_2 and X_3 are the coded factors, which are related to the
 102 actual factors x_1 , x_2 and x_3 in Table 1 by equation (4):

$$103 \quad X_i = \frac{x_i - x_o}{\Delta x} \quad (4)$$

104 where:

105 X_i = coded value for the i th input (that is, X_i),

106 x_o = mid value for the experimental design, and

107 $\Delta x = (x_{high} - x_o) = (x_o - x_{low})$.

108 The terms X_{high} and X_{low} represent the chosen upper and lower design limits, respectively.

109 Table 1: Coded and uncoded levels of variables for the RSM Box–Behnken design.

Variable	Symbol	Coded factor levels		
		-1	0	+1
Oil bath Temperature ($^{\circ}\text{C}$)	x_1	120	135	150
Time (min)	x_2	15	37.5	60
[NaOH] (g/l)	x_3	10	30	50

110 **2.3. Acid value determination**

111 About 1 g of the crude terephthalic acid obtained from each of the alkaline solvolysis runs was accurately
 112 weighed into a 100 ml beaker containing 25 ml of pyridine. The mixture was stirred till the sample

113 completely dissolved, after which 25 ml of water and 2 – 3 drops of phenolphthalein indicator were
 114 added. The solution was titrated against 0.5 M potassium hydroxide solution till a permanent pink end
 115 point was obtained. A blank determination was also carried out, excluding the sample. The acid value was
 116 determined from the formula:

$$117 \quad AV \text{ (mg KOH/g)} = \frac{56.1 \times M \times (V_S - V_B)}{w} \quad (5)$$

118 Where M is the molarity of the KOH solution (mol/dm³), V_S and V_B are the titre values of sample and
 119 blank, respectively, and w (g) is the mass of sample taken for test.

120 **2.4. Instrumental analysis**

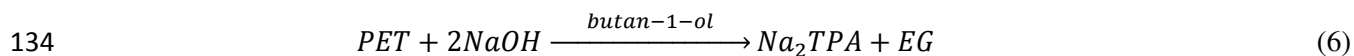
121 Differential thermal analysis of the solid product was carried out on a Netzsch Differential Thermal
 122 Analyzer (Model DTA 404 PC Eos) at a heating rate of 10 °C/min. Infrared spectroscopy was carried out
 123 on a Thermo Nicolet iS5 FT-IR equipped with iD3 Attenuated Total Reflectance (ATR) accessory and
 124 Omnic FTIR software for spectra processing and analysis.

125 The analysis of the liquid products was done on an Agilent 6890/5973 GCMS System with 5% phenyl
 126 methyl siloxane capillary column. Helium was used as the carrier gas at a flow rate of 1.5 ml/min. The
 127 oven temperature was programmed as follows: 35 °C for 5 min, then 4 °C/min to 150 °C for 2 min, and
 128 finally at 20 °C/min to 250 °C for 5 min. The quadrupole temperature was set at 150°C. Methanol was
 129 used as solvent.

130 **3. Results and Discussion**

131 **3.1. PET depolymerization kinetics**

132 The overall chemical equation for the alkaline solvolysis of PET to yield disodium terephthalate and
 133 ethylene glycol may be written as shown:



135 The kinetics of PET depolymerization in alkaline butan-1-ol was studied by assuming that the reaction is
 136 irreversible homogeneous second order with respect to PET and NaOH. The second order homogenous
 137 reaction kinetic model is given by (Lopez-Fonseca *et al*, 2008):

$$138 \quad -\frac{dC_{PET}}{dt} = kC_{PET}C_{NaOH} \quad (7)$$

139 where C_{PET} and C_{NaOH} represent the quantity of PET and NaOH present per unit volume of the reaction
 140 mixture at any given time, respectively.

141 If the initial amount of PET is given to be (C_{PET})_o, then it can be said that:

$$142 \quad C_{PET} = (C_{PET})_o(1 - X_{PET}) \quad (8)$$

143 where X_{PET} is the fractional conversion of PET, which is given by:

$$144 \quad X_{PET} = \frac{W_o - W_f}{W_o} \quad (9)$$

145 where W_o and W_f are the initial and final masses of the PET samples, respectively.

146 Equation (7) can be written for the chemical reaction in (6) as:

$$147 \quad \frac{dX_{PET}}{dt} = k(C_{PET})_o(1 - X_{PET})(M - 2X_{PET}) \quad (10)$$

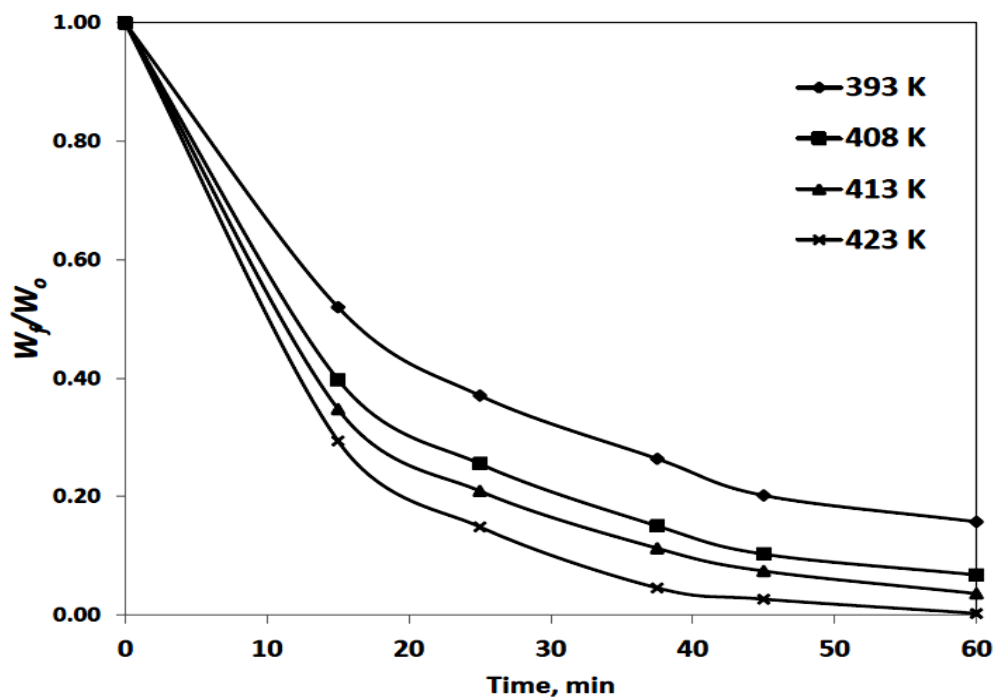
148 where M is the initial NaOH to PET molar ratio. If it is assumed that the volume of the reaction mixture is
149 constant, the integration of equation (10) gives:

$$150 \quad \left(\frac{1}{M-2}\right) \ln \left[\frac{M-2X_{PET}}{M(1-X_{PET})}\right] = k(C_{PET})_o t \quad (11)$$

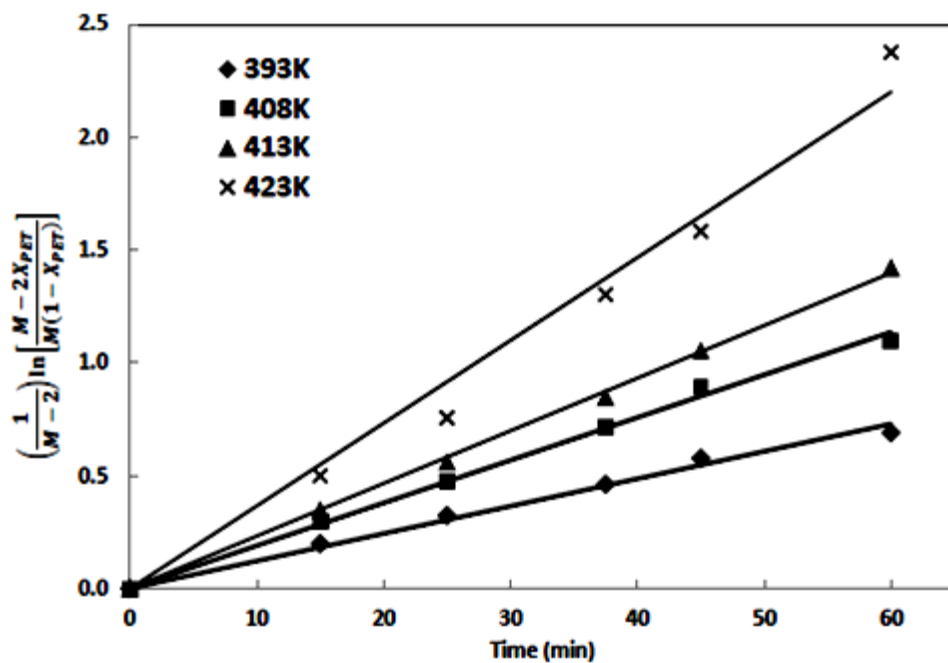
151 where k is the rate constant.

152 The PET dissolution kinetics plots are presented in Figure 1 with the initial mass of PET and the initial
153 concentration of sodium hydroxide kept constant at 5 g and 40 g/l, respectively (NaOH:PET molar ratio,
154 $M = 3.84$) while the temperature of the oil bath was varied from 120 – 150 °C (393 – 423 K).

155 A plot of $\left(\frac{1}{M-2}\right) \ln \left[\frac{M-2X_{PET}}{M(1-X_{PET})}\right]$ against t gives straight lines with zero intercepts as shown in Figure 2
156 while the numerical values of depolymerization rate constants at different oil bath temperatures are
157 presented in Table 2. Each of the plots was found to fit the kinetic model presented in equation (12) with
158 R^2 values of 0.98, or better.



159
 160 Figure 1 Alkaline Solvolysis of PET in 40 g/l NaOH in Butan-1-ol at different oil bath
 161 temperatures
 162



163
 164 Figure 2: Fitting of kinetic data according to equation (10) for M = 3.84 at different oil bath
 165 temperatures
 166

167
168
169

Table 2: Kinetic Data Obtained for the alkaline solvolysis of PET

Temperature of Oil bath, T (K)	Apparent rate constant, $k(C_{\text{PET}})_0$ ($\text{min}^{-1}) \times 10^3$	R^2 value
393	12.1	0.9904
408	18.9	0.9962
413	23.3	0.9987
423	36.7	0.9808

170 .

171 3.2. Analysis of PET alkaline solvolysis products

172 From the theory of PET alkaline solvolysis, the expected primary product is terephthalic acid. For each
173 experimental run, a white powder was obtained and this was subjected to various physical and chemical
174 tests.

175 3.2.1. Acid value

176 The acid values of the solid products obtained from the alkaline solvolysis of PET in the alcoholic media
177 under the different conditions studied gave an average of 670.6 mg KOH/g for the butan-1-ol media.
178 Comparing this average value with the theoretical acid number of TPA (that is, 675 mgKOH/g), it can be
179 said that the observed variations might have been a result of impurities and other reaction perturbations.

180 3.2.2. FTIR analysis

181 The FTIR spectrum of the product obtained from the decomposition of PET using 40 g/l NaOH in the
182 alcohol is shown in Figure 3. A look at the IR spectra indicated that each product is a *p*-substituted
183 aromatic compound, considering the absorption bands at 1600 and 1420 cm^{-1} ($-\text{C}-\text{C}-$ stretch for aromatic
184 compounds), and the sharp absorption band around 750 cm^{-1} . The very broad $-\text{OH}$ band occurring
185 between 3000 and 3500 cm^{-1} , and the $-\text{C}=\text{O}$ band around 1700 cm^{-1} is an indication that the product is a
186 carboxylic acid. The IR spectrum for terephthalic acid is shown in Figure 4 for comparison.

187 3.2.3. Thermal analysis of the solid products

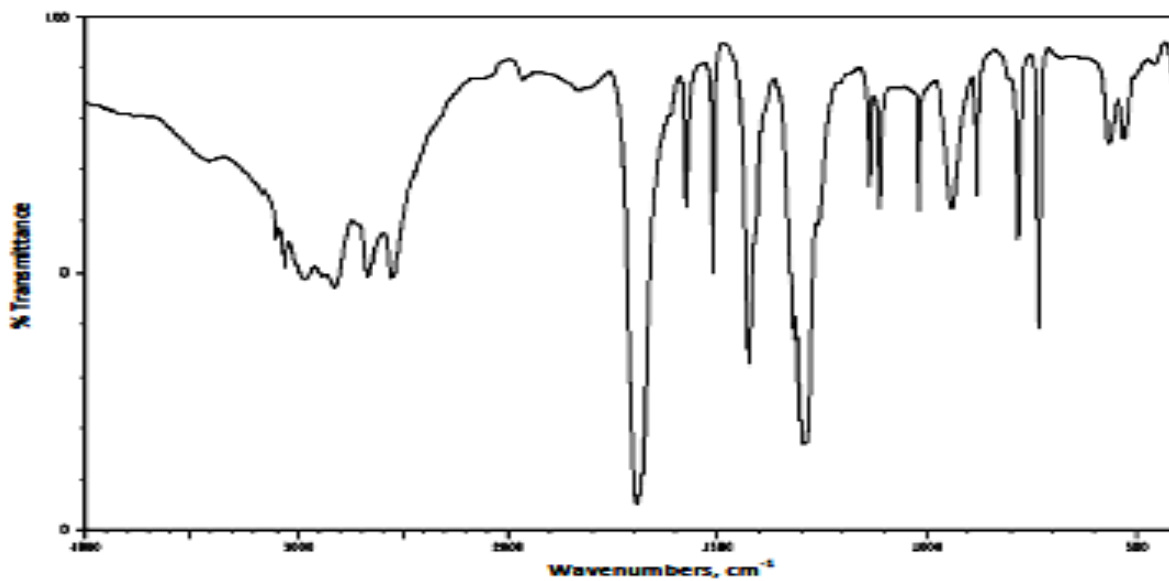
188 The DTA scans for the solid product obtained from the alkaline solvolysis runs show that its melting
189 point is above 380 °C (Figure 5). The melting point of terephthalic acid is between 300 and 402 °C, while
190 that of PET is around 260 °C. Since the thermograph in Figure 5 does not show any endothermic peak
191 around 260 °C, it can be concluded that solid product is different from PET. In addition, the high melting
192 points are close to that of terephthalic acid. However, the appearance of additional endothermic peaks at
193 122.1, 210.8 and 297 °C suggests the formation of other products other than terephthalic acid.



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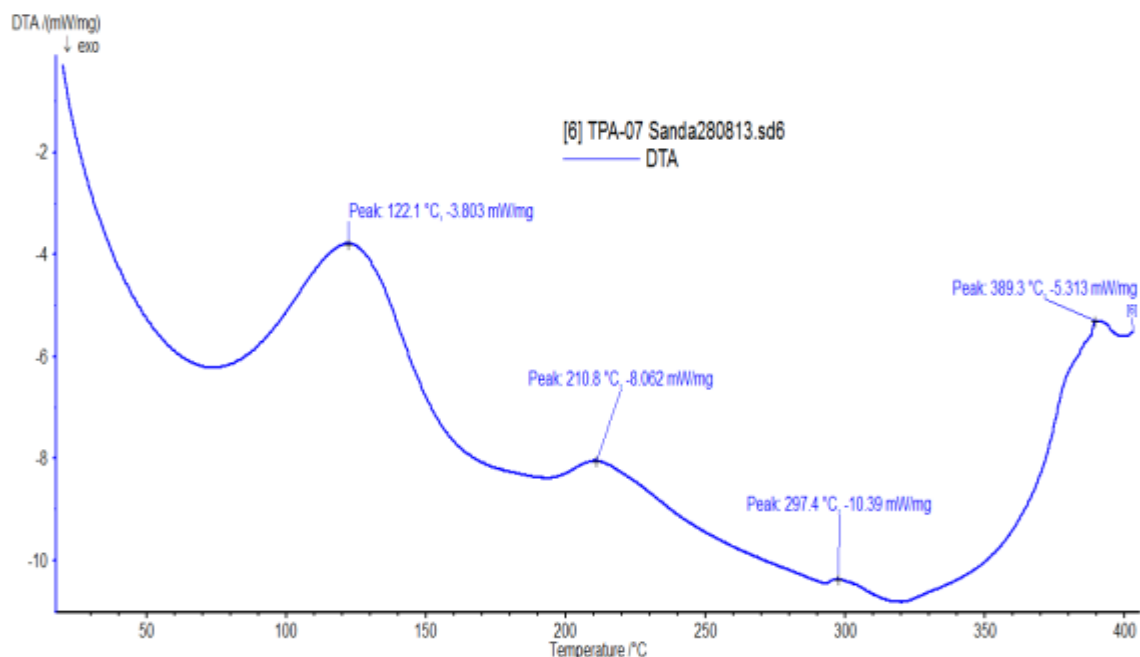
Figure 3: IR spectrum of the solid product



196

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Figure 4: IR Spectrum of pure terephthalic acid



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199 Figure 5: DTA Thermograph for the solid Product

200 **3.3. PET Solvolytic Decomposition study Using Response Surface Methodology**

201 The relationship between the responses (% decomposition of PET and terephthalic acid yield) and three
 202 independent variables (reaction time, temperature and alkali concentration) were studied in order to
 203 optimize the alkaline solvolysis of PET. The design matrix in actual terms and the experimental results of
 204 RSM are presented in Table 3. The experimental data were fitted to Equation (3) to obtain mathematical
 205 regression quadratic models for the relationship between the responses (% decomposition of PET and
 206 TPA yield) and the experimental factors:

207
$$Y_{PET} = 89.067 + 2.425X_1 + 7.075 X_2 + 19.550 X_3 - 5.058 X_1^2 - 4.158 X_2^2 - 12.008 X_3^2 +$$

 208
$$1.350X_1X_2 + 4.100X_1X_3 - 2.700X_2X_3 \quad (R^2 = 0.9902)$$

 209 (13)

210
$$Y_{TPA} = 0.73925 + 0.02013X_1 + 0.05872 X_2 + 0.16226 X_3 - 0.04198 X_1^2 - 0.03451 X_2^2 -$$

 211
$$0.09967 X_3^2 + 0.01120X_1X_2 + 0.03403X_1X_3 - 0.02241X_2X_3 \quad (R^2 = 0.9900)$$

 212 (14)

213 where Y_{PET} and Y_{TPA} represent the percentage decomposition of PET and TPA yield, respectively.

214 ANOVA for studying the significance of fit from the quadratic equations for the experimental data is
 215 shown in Tables 4 and 5, with P-values lower than 0.05 indicating significant model terms. In addition,

216 the lack-of-fit for the model is insignificant with a P-value of 0.846 for PET decomposition and 0.844 for
 217 TPA yield, indicating that the models are suitable for fitting the experimental data. The quadratic models
 218 are significant ($P < 0.001$), accounting for over 98 % of the observations. The extent of PET
 219 decomposition depends on the process temperature and the concentration of NaOH for the alcohol
 220 studied.

221 Table 3: Actual and Predicted Responses for the Alkaline Solvolysis of PET in Butan-1-ol Media

Run	Temperature (°C)	Time (min)	[NaOH], g/L	% Decomposition of PET		TPA yield (g/g PET)	
				Actual	Predicted	Actual	Predicted
1	120	15	30	70.6	71.70	0.586	0.595
2	150	15	30	72.6	73.85	0.603	0.613
3	120	60	30	84.4	83.15	0.701	0.690
4	150	60	30	91.8	90.70	0.762	0.753
5	120	37.5	10	54.4	54.12	0.452	0.449
6	150	37.5	10	51.2	50.77	0.425	0.421
7	120	37.5	50	84.6	85.03	0.702	0.706
8	150	37.5	50	97.8	98.08	0.812	0.814
9	135	15	10	44.4	43.57	0.369	0.362
10	135	60	10	61.6	63.13	0.511	0.524
11	135	15	50	89.6	88.08	0.744	0.731
12	135	60	50	96.0	96.83	0.797	0.804
13	135	37.5	30	84.6	89.07	0.702	0.739
14	135	37.5	30	91.4	89.07	0.759	0.739
15	135	37.5	30	91.2	89.07	0.757	0.739

222

223

224 Table 4: ANOVA for the Response Model for the % PET Decomposition in Butan-1-ol Media

Source	Df ^a	SS ^b	MS ^c	F	P
Model	9	4236.98	470.78	56.02	<0.001
Temperature (°C) (X ₁)	1	47.04	47.04	5.60	0.064
Time (min) (X ₂)	1	400.44	400.44	47.65	0.001
[NaOH], g/L (X ₃)	1	3057.62	3057.62	363.86	<0.001
X ₁ ²	1	94.47	94.47	11.24	0.020
X ₂ ²	1	63.85	63.85	7.60	0.040
X ₃ ²	1	532.43	532.43	63.36	0.001
X ₁ X ₂	1	7.29	7.29	0.87	0.394
X ₁ X ₃	1	67.24	67.24	8.00	0.037
X ₂ X ₃	1	29.16	29.16	3.47	0.122
Lack of fit	2	12.07	4.02	0.27	0.846
Pure error	3	29.95	14.97		
Total	14				

225 *a: Degrees of freedom b: Sum of squares*

c: Mean squares

226

227 Table 5: ANOVA for the Response Model for TPA yield

Source	Df ^a	SS ^b	MS ^c	F	P
Model	9	0.2917	0.0324	54.98	< 0.001
Temperature (°C) (X ₁)	1	0.0032	0.0032	5.50	0.066
Time (min) (X ₂)	1	0.0275	0.0275	46.63	0.001
[NaOH], g/L (X ₃)	1	0.2106	0.2106	357.20	<0.001
X ₁ ²	1	0.0039	0.0065	11.00	0.021
X ₂ ²	1	0.0027	0.0044	7.42	0.042
X ₃ ²	1	0.0367	0.0367	62.21	0.001
X ₁ X ₂	1	0.0005	0.0005	0.82	0.406
X ₁ X ₃	1	0.0047	0.0047	7.96	0.037
X ₂ X ₃	1	0.0020	0.0020	3.36	0.126
Lack of fit	3	0.0009	0.0003	0.27	0.844
Pure error	2	0.0021	0.0010		
Total	14				

228 *a: Degrees of freedom b: Sum of squares c: Mean squares*

229 The response surface 3D and contour plots for PET decomposition in relation to temperature, sodium
 230 hydroxide concentration and reaction time are illustrated in Figures 6 and 7, with sodium hydroxide
 231 concentration, time and temperature kept at their mid-point levels in (a), (b) and (c), respectively. From
 232 the studies, it was found that temperature and alkali concentration play an important role in the alkaline
 233 solvolysis of PET in butan-1-ol media.

234 The optimum PET decomposition and terephthalic acid yield for the conditions under study were found
 235 to be 99.85 % and 0.8289 g TPA/g PET, respectively for an optimum temperature, NaOH concentration
 236 and reaction time of 139.1 °C, 45.13 g/L and 50.71 min, respectively as shown by the Minitab 16
 237 Optimization plots in Figure 8.

238 3.4. Analysis of the liquid product by gas chromatography

239 The chromatograph for the filtrate obtained from the solvolysis run is shown in Figure 9. From the theory
 240 of the alkaline solvolysis process, the liquid phase obtained at the end of each run is expected to contain
 241 ethylene glycol and the alcohol used. Most of the compounds identified from the chromatograph are
 242 actually presented in form of combinations of the actual compounds of interest due to the conditions
 243 specified for the analyses (up to 300 °C).

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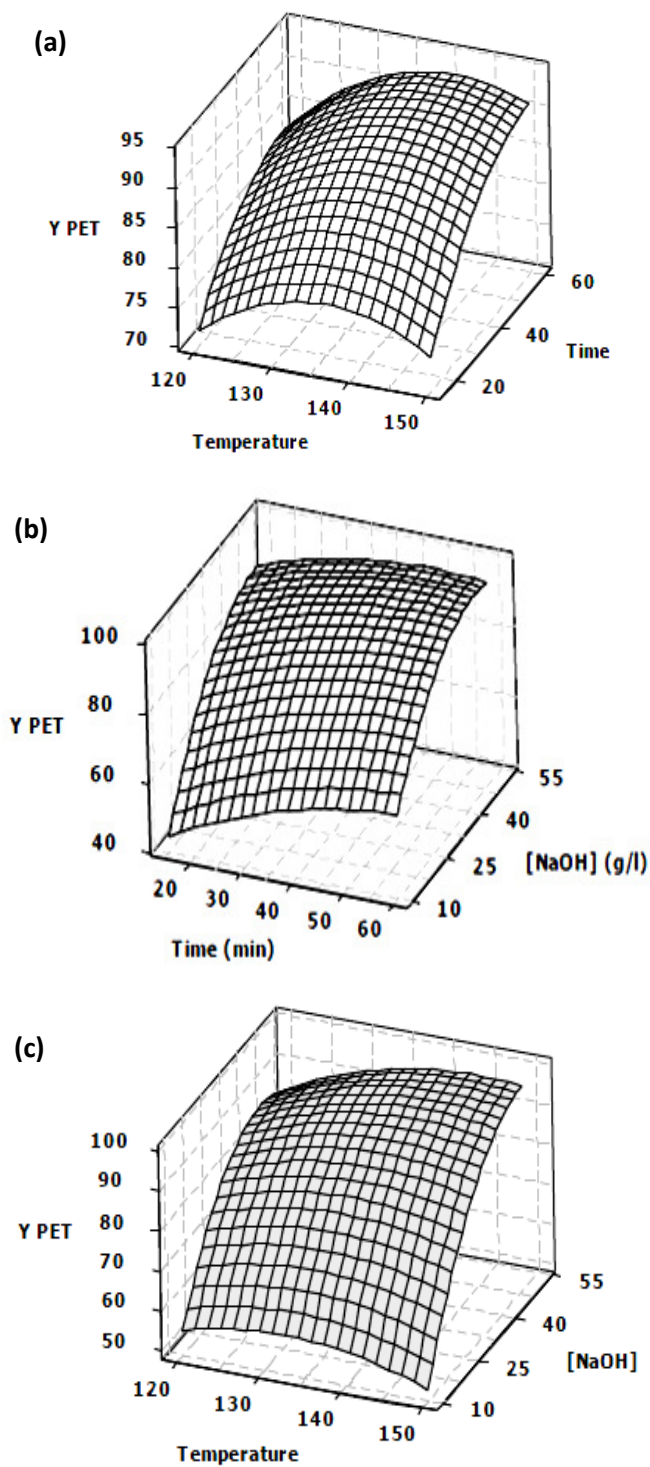


Figure 6: Response surface 3D plot for % decomposition of PET.

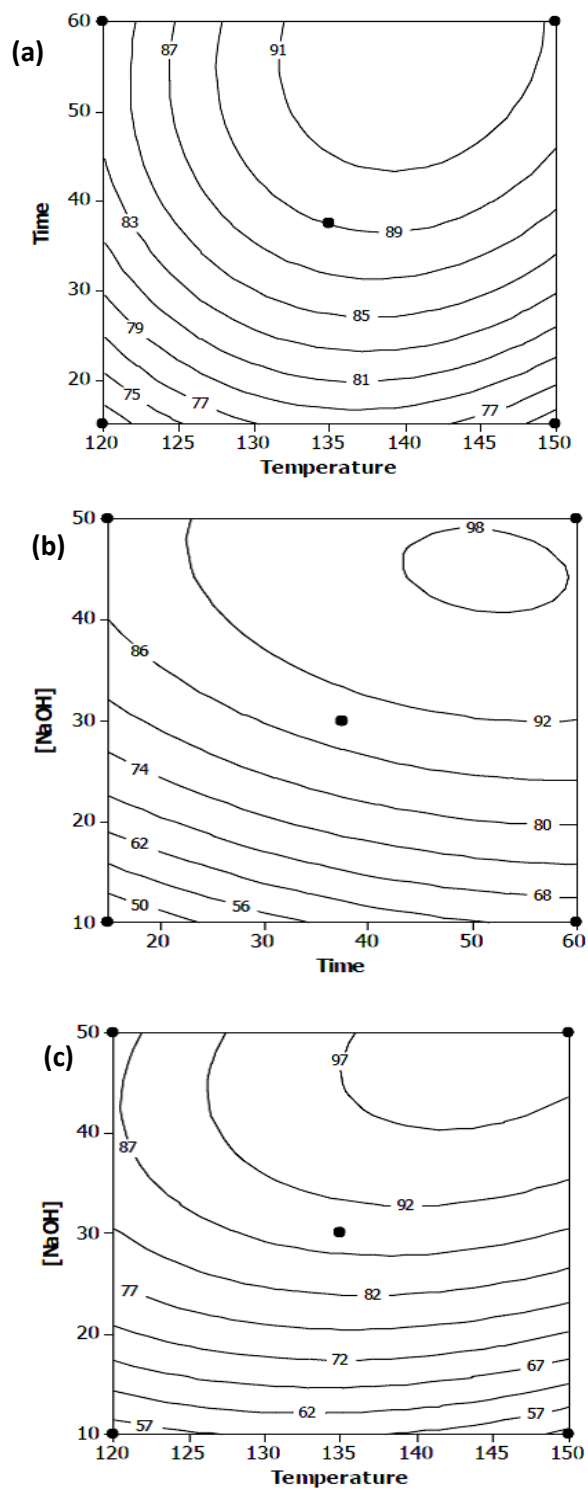
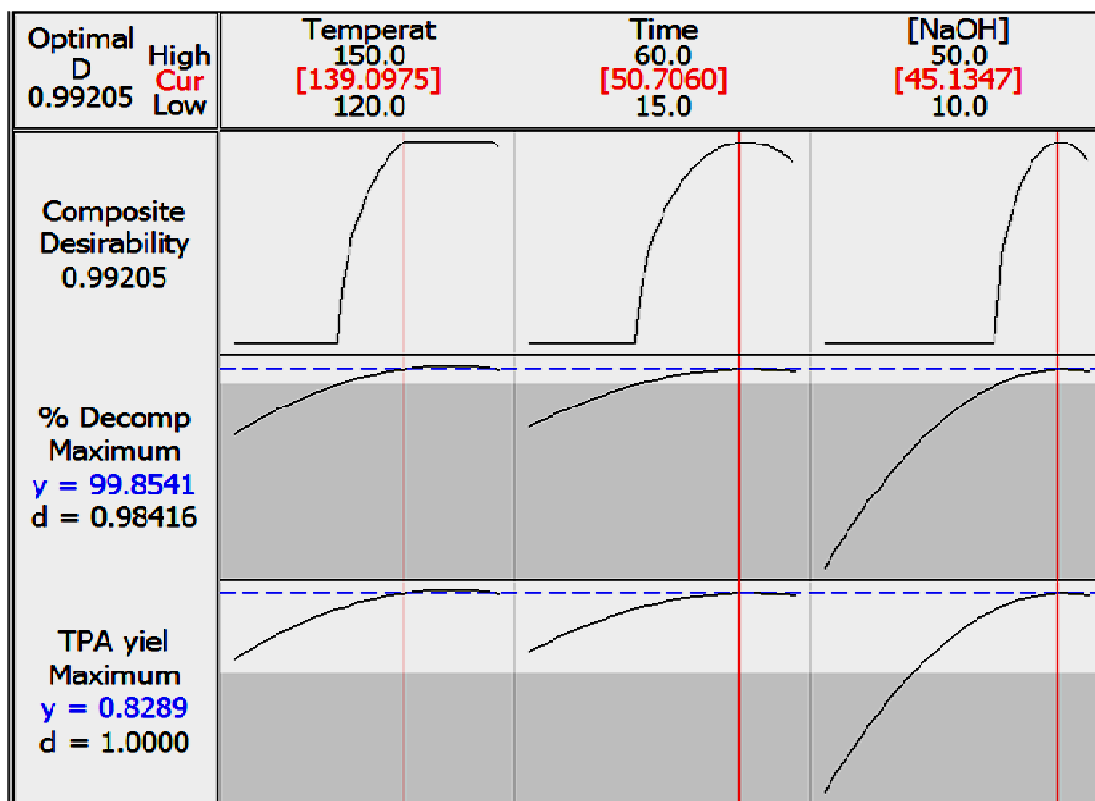


Figure 7: Response surface contour plot for % decomposition of PET.

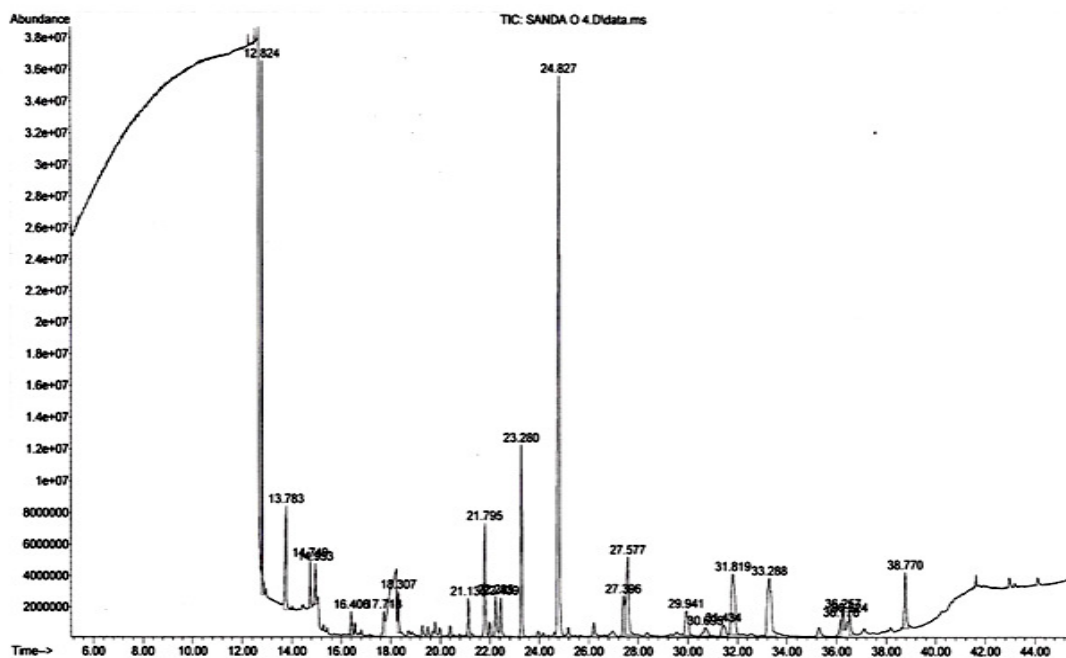


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Figure 8: Optimization plots for the alkaline solvolysis of PET for the conditions under study



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Figure 9: Gas chromatograph for the filtrate obtained from the alkaline solvolysis of PET in butan-1-ol media

267 **4. Conclusion**

268 This study has shown that alkaline solvolysis of PET is a simple and effective thermo-chemical route for
269 processing postconsumer PET into value added products. The rate of decomposition of PET depends on
270 the concentration of alkali, process time and temperature, with a predicted optimum conversion of over 99
271 % within an hour. Chemical analyses showed that the solid products obtained have properties similar to
272 those of terephthalic acid while the liquid products contain ethylene glycol in addition to the solvent used,
273 along with various products of side reactions, implying that postconsumer PET can be a good source of
274 terephthalic acid, ethylene glycol and other value-added products.

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