



Alkaline Solvolysis of Poly(Ethylene Terephthalate) in Butan-1-ol Media: Kinetics and Optimization Studies

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Authors' contributions

This work was carried out in collaboration between all authors. Author OS designed the study, performed the statistical analysis, wrote the protocol and wrote the first draft of the manuscript. Authors EAT and GAO managed the analyses of the study. Author OS managed the literature searches. All authors read and approved the final manuscript.

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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.

Keywords: Polyethylene terephthalate; Butan-1-ol; terephthalic acid; alkaline solvolysis.

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1. INTRODUCTION

Poly(ethylene terephthalate) (PET) is a linear thermoplastic, bluish-white resin made from terephthalic acid and ethylene glycol through poly-condensation [1]. It is an indispensable material owing to its low cost [2], excellent tensile strength, chemical resistance, clarity, processability, and reasonable thermal stability [3]. 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 [4–6]. 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 [7].

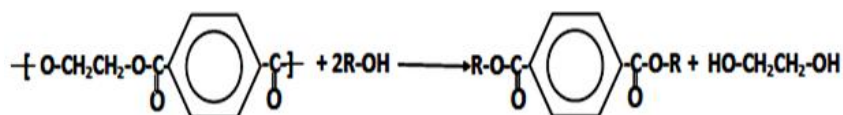
Combining the relative simplicity of the polymerization process with the durable mechanical properties of PET, industries throughout the world have used PET as the staple polymer for beverage packaging. Along with this widespread use of PET is the inevitable creation of large amounts of post-consumer 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 [8]. With the increase in the amount of PET wastes, its disposal began to pose serious economical and environmental problems. The recycling of PET does not only serve as a partial solution to the solid waste problem but also contributes to the conservation of raw petrochemical products and energy. Products made from recycled plastics can result in 50-60% capital saving as compared to making the same product from virgin resin [9].

Recycling is assumed to be one of the best approaches to solve the problems of PET wasted accumulation. Various methods such as primary recycling [10,11], mechanical recycling [12] and chemical recycling [10,13–15] have been considered to recycle the PET. Among the various methods of PET recycling, only chemical recycling conforms to the principles of sustainable development because it has potentials for yielding the raw materials from which PET is originally made. However, the various chemical recycling routes reported in various works (such as hydrolysis, alcoholysis and aminolysis) are fraught with shortcomings such as the need for high pressure and corrosion resistant equipment [16], high temperature [13], and long reaction times [14]. Taking into account the cost and energy consumption, chemical reclamation methods are the most effective recycling methods for PET. Alkaline solvolysis was chosen for this study because it operates under less hazardous conditions, effectively eliminating the need for corrosion resistant pressure vessels (since it can be carried out at atmospheric pressure).

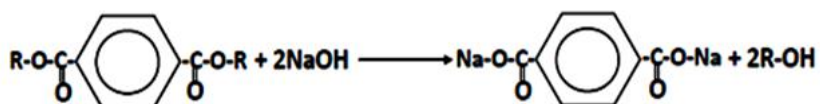
Preliminary studies on the alkaline solvolysis of postconsumer PET in methanol or ethanol media showed that it is possible to decompose PET according to the reaction mechanisms shown below [17,18]:

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

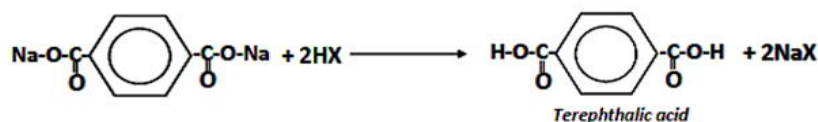


This step is the alcoholysis step, similar to the methanolysis process reported by Genta et al. [16].

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



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



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

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

2. EXPERIMENTAL

2.1 Materials

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

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

2.2 Alkaline Solvolysis of PET

For each run, about 5 g of PET flakes was placed in a two-neck 500 cm³ flask with 100 cm³ of 40 g.dm⁻³ sodium hydroxide in butan-1-ol. The flask was dipped in a thermostatic oil bath with the temperature of the bath maintained between 120 and 150 °C. The mixture was heated for 10 – 60 min under reflux and at the end of each run, 100 cm³ of distilled water was added to dissolve the solid products. The mixture was then filtered

through a Whatman No. 1 filter paper to separate the unreacted PET from the mother liquor and the residue was washed with additional 50 cm³ of distilled water. Terephthalic acid (TPA) was precipitated from the filtrate by the addition of 1 mol.dm⁻³ hydrochloric acid and removed with a Whatman No. 1 filter paper. The unreacted PET and the TPA produced were dried in an oven for 2 h at 105 °C and weighed. The percent decomposition of PET was determined by gravimetry using the expression below:

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

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

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

In order to optimize the alkaline solvolysis process, a three-level-three factor Box-Behnken design was employed for this study, with 15 experimental runs per alcohol. The factors investigated in this study were temperature (°C), reaction time (min) and alkali concentration (g.dm⁻³). The coded and uncoded levels of the independent factors are shown in Table 1.

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

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

Where Y is the predicted response (% PET decomposition or TPA yield), δ_o is the intercept

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 (°C)	x_1	120	135	150
Time (min)	x_2	15	37.5	60
[NaOH] (g.dm ⁻³)	x_3	10	30	50

term, δ_1 , δ_2 , 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 quadratic coefficients. In addition, the terms X_1 , X_2 and X_3 are the coded factors, which are related to the actual factors x_1 , x_2 and x_3 in Table 1 by equation (4):

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

Where,

X_i = coded value for the i th input (that is, X_i),
 x_o = mid value for the experimental design, and
 $\Delta x = (x_{high} - x_o) = (x_o - x_{low})$.

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

2.3 Acid Value Determination

About 1 g of the crude terephthalic acid obtained from each of the alkaline solvolysis runs was accurately weighed into a 100 cm³ beaker containing 25 cm³ of pyridine. The mixture was stirred till the sample completely dissolved, after which 25 cm³ of water and 2 – 3 drops of phenolphthalein indicator were added. The solution was titrated against 0.5 mol.dm⁻³ potassium hydroxide solution till a permanent pink end point was obtained. A blank determination was also carried out, excluding the sample. The acid value was determined from the formula:

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

Where M is the molarity of the KOH solution (mol.dm⁻³), V_s and V_B are the titre values of sample and blank, respectively, and w (g) is the mass of sample taken for test.

2.4 Instrumental Analysis

Differential thermal analysis of the solid product was carried out on a Netzsch Differential Thermal Analyzer (Model DTA 404 PC Eos) at a heating rate of 10°C/min. Infrared spectroscopy was carried out on a Thermo Nicolet iS5 FT-IR equipped with iD3 Attenuated Total Reflectance

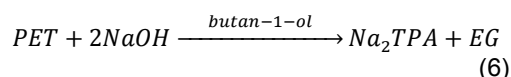
(ATR) accessory and Omnic FTIR software for spectra processing and analysis.

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

3. RESULTS AND DISCUSSION

3.1 PET Depolymerization Kinetics

The overall chemical equation for the alkaline solvolysis of PET to yield disodium terephthalate (Na₂TPA) and ethylene glycol (EG) may be written as shown:



The kinetics of PET depolymerization in alkaline butan-1-ol was studied by assuming that the reaction is irreversible homogeneous second order with respect to PET and NaOH. The second order homogenous reaction kinetic model is given by Lopez-Fonseca et al. [19]:

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

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

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

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

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

$$X_{PET} = \frac{w_o - w_f}{w_o} \quad (9)$$

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

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

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

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

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

where k is the rate constant.

The PET dissolution kinetics plots are presented in Fig. 1 with the initial mass of PET and the initial concentration of sodium hydroxide kept constant at 5 g and 40 g.dm⁻³, respectively (NaOH: PET molar ratio, $M = 3.84$) while the temperature of the oil bath was varied from 120–150°C (393 – 423 K).

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 Fig. 2 while the numerical values of depolymerization rate constants at different oil bath temperatures are presented in Table 2. Each of the plots was found to fit the kinetic model presented in equation (11) with R^2 values of 0.98, or better.

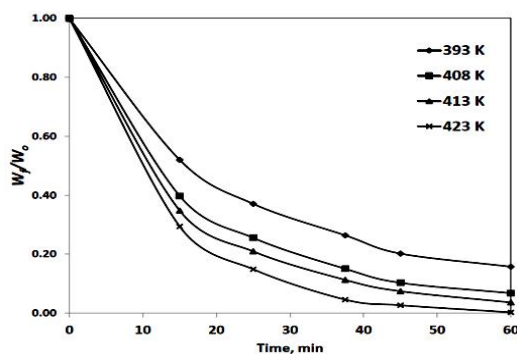


Fig. 1. Alkaline solvolysis of PET in 40 g.dm⁻³ NaOH in Butan-1-ol at different oil bath temperatures

3.2 Analysis of PET Alkaline Solvolysis Products

From the theory of PET alkaline solvolysis, the expected primary product is terephthalic acid.

For each experimental run, a white powder was obtained and this was subjected to various physical and chemical tests.

3.2.1 Acid value

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

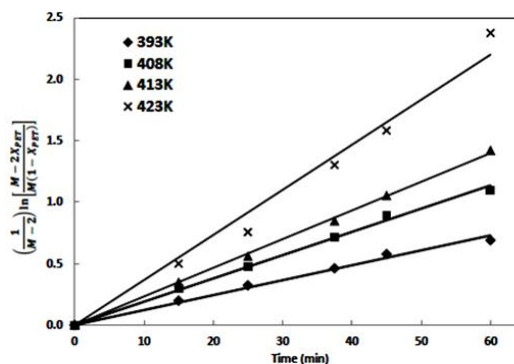


Fig. 2. Fitting of kinetic data according to equation (10) for $M = 3.84$ at different oil bath temperatures

Table 2. Kinetic data obtained for the alkaline solvolysis of PET

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

3.2.2 FTIR analysis

The FTIR spectrum of the product obtained from the decomposition of PET using 40 g.dm⁻³ NaOH in the alcohol is shown in Fig. 3. A look at the IR spectra indicated that each product is a *p*-substituted aromatic compound, considering the absorption bands at 1600 and 1420 cm⁻¹ (–C–C– stretch for aromatic compounds), and the sharp absorption band around 750 cm⁻¹. The very broad –OH band occurring between

3000 and 3500 cm^{-1} , and the -C=O band around 1700 cm^{-1} is an indication that the product is a carboxylic acid. The IR spectrum for terephthalic acid is shown in Fig. 4 for comparison.

3.2.3 Thermal analysis of the solid products

The DTA scans for the solid product obtained from the alkaline solvolysis runs show that its melting point is above 380°C (Fig. 5). The

melting point of terephthalic acid is between 300 and 402°C, while that of PET is around 260°C [20]. Since the thermograph in Fig. 5 does not show any endothermic peak around 260°C, it can be concluded that solid product is different from PET. In addition, the high melting points are close to that of terephthalic acid. However, the appearance of additional endothermic peaks at 122.1, 210.8 and 297°C suggests the formation of other products other than terephthalic acid.

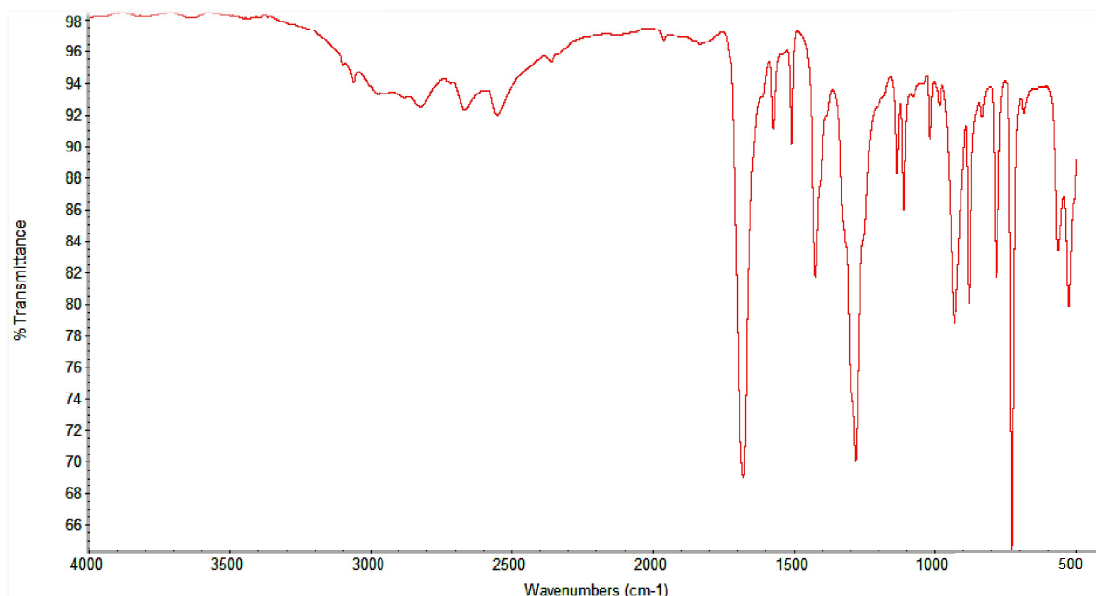


Fig. 3. IR spectrum of the solid product

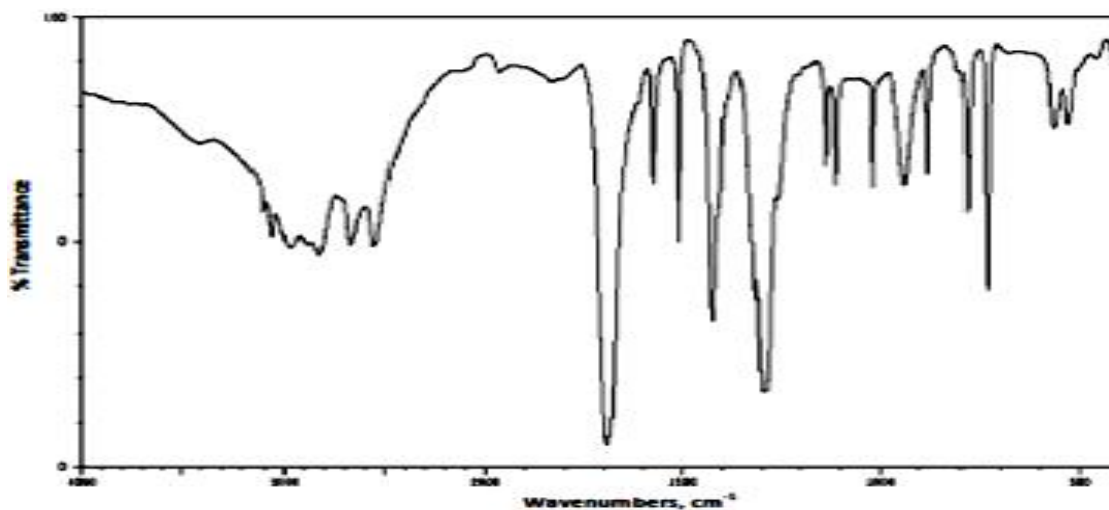


Fig. 4. IR Spectrum of pure terephthalic acid

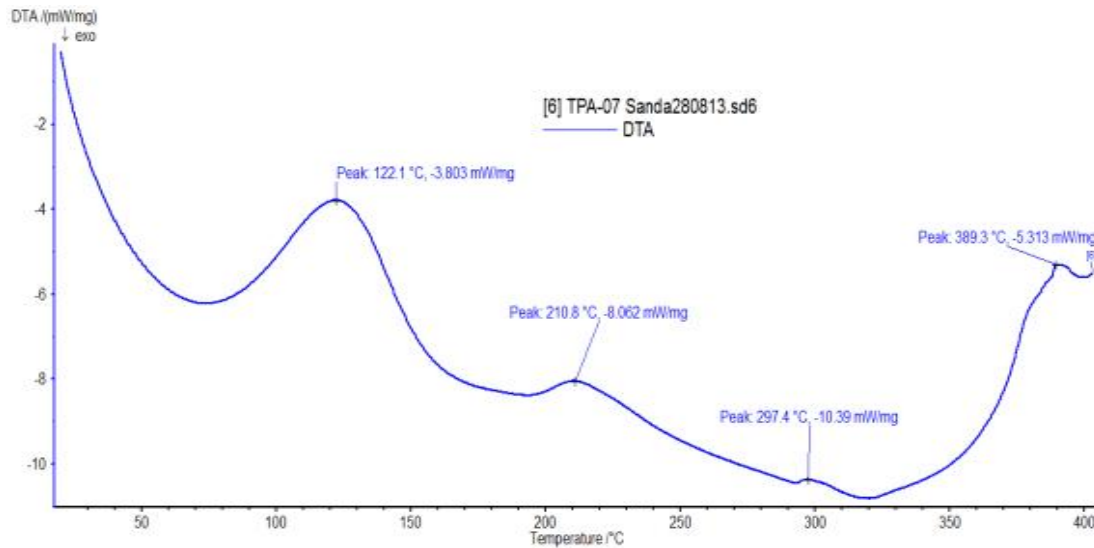


Fig. 5. DTA thermograph for the solid product

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

3.3 PET Solvolytic Decomposition Study Using Response Surface Methodology

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

$$Y_{\text{PET}} = 89.067 + 2.425X_1 + 7.075 X_2 + 19.550 X_3 - 5.058 X_1^2 - 4.158 X_2^2 - 12.008 X_3^2 + 1.350X_1X_2 + 4.100X_1X_3 - 2.700X_2X_3 \quad (R^2 = 0.9902) \quad (12)$$

$$Y_{\text{TPA}} = 0.73925 + 0.02013X_1 + 0.05872 X_2 + 0.16226 X_3 - 0.04198 X_1^2 - 0.03451 X_2^2 - 0.09967 X_3^2 + 0.01120X_1X_2 + 0.03403X_1X_3 - 0.02241X_2X_3 \quad (R^2 = 0.9900) \quad (13)$$

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

ANOVA for studying the significance of fit from the quadratic equations for the experimental data is shown in Tables 4 and 5, with P-values lower than 0.05 indicating significant model terms. In addition, the lack-of-fit for the model is insignificant with a P-value of 0.846 for PET decomposition and 0.844 for TPA yield, indicating that the models are suitable for fitting the experimental data. The quadratic models are significant ($P < 0.001$), accounting for over 98% of the observations. The extent of PET decomposition depends on the process temperature and the concentration of NaOH for the alcohol studied.

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

The optimum PET decomposition and terephthalic acid yield for the conditions under study were found to be 99.85% and 0.8289 g TPA/g PET, respectively for an optimum temperature, NaOH concentration and reaction time of 139.1°C, 45.13 g.dm⁻³ and 50.71 min, respectively as shown by the Minitab 16 Optimization plots in Fig. 8.

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_1X_2	1	7.29	7.29	0.87	0.394
X_1X_3	1	67.24	67.24	8.00	0.037
X_2X_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				

a: Degrees of freedom, b: Sum of squares, c: Mean squares

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_1X_2	1	0.0005	0.0005	0.82	0.406
X_1X_3	1	0.0047	0.0047	7.96	0.037
X_2X_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				

a: Degrees of freedom, b: Sum of squares, c: Mean squares

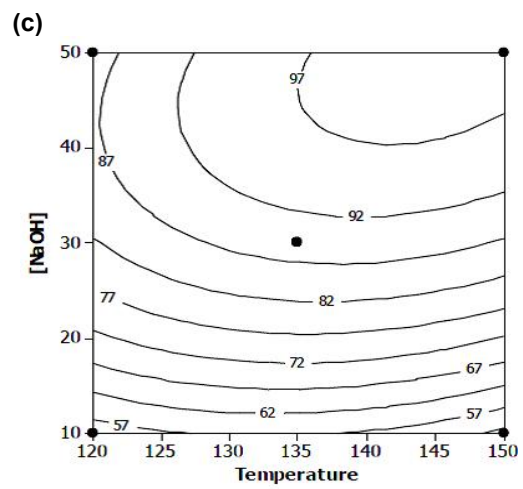
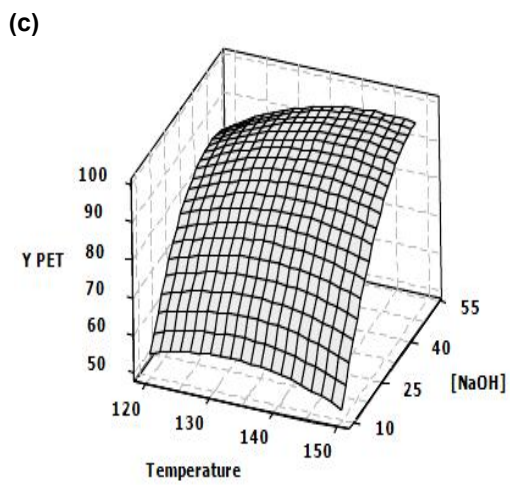
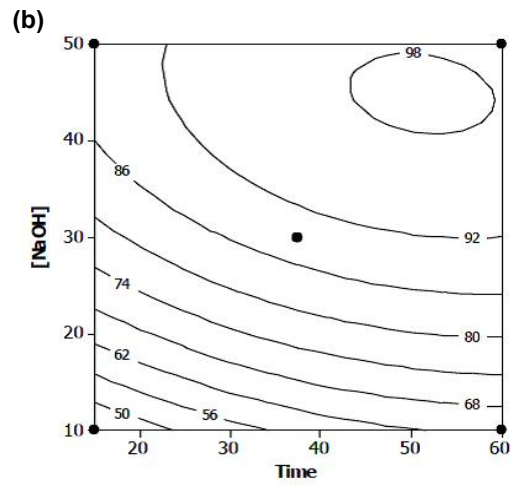
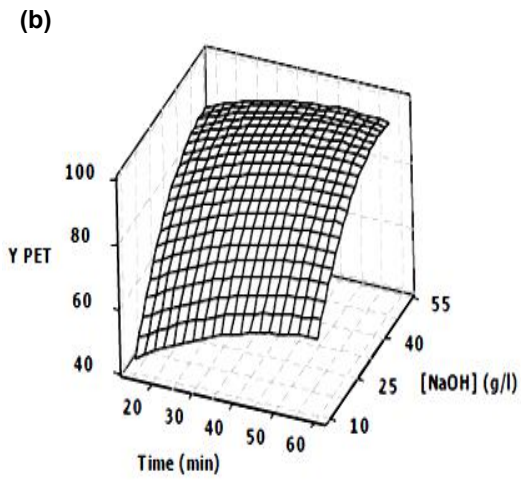
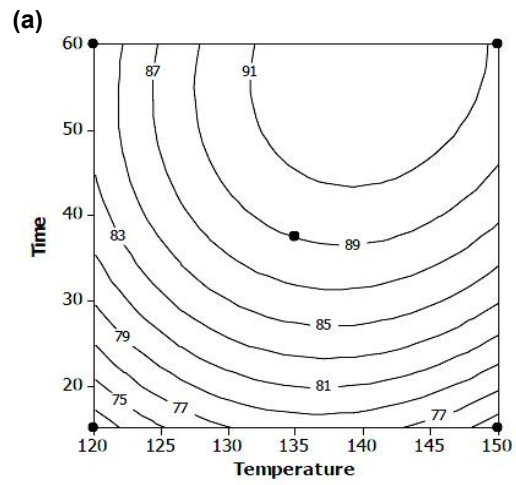
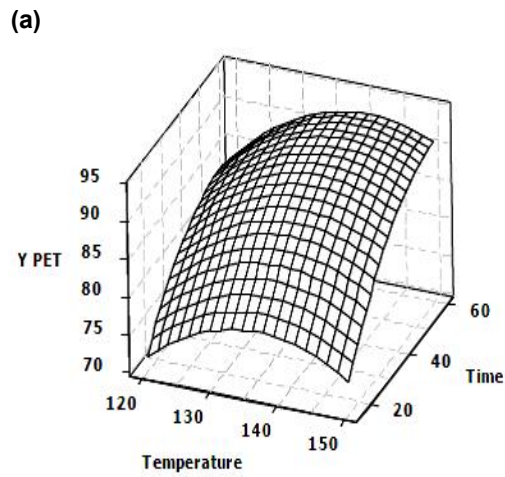


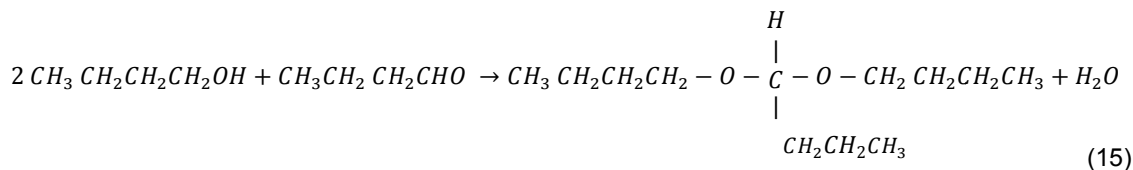
Fig. 6. Response surface 3D plot for % decomposition of PET

Fig. 7. Response surface contour plot for % decomposition of PET

3.4 Analysis of the Liquid Product by Gas Chromatography

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

The gas chromatograph for the liquid product obtained from the alkaline solvolysis run shows that in addition to ethylene glycol, various products were formed as a result of series of chemical reactions such as chain elongation, formation of glycol ethers or rearrangement reactions (Table 6). Since the reactions were carried in the presence of air, there is a possibility of the partial oxidation of butan-1-ol to give butanal, which further combined with butan-1-ol to give 1,1-dibutoxy butane according to the reactions below:



The reaction (15) would normally occur in the presence of an anhydrous acid such as hydrogen chloride [21] which probably came from the acid used in precipitating the terephthalic acid. Some of the compounds (for example, butoxy-2,4-dimethyl-1-pentene and 2-Ethylnon-1-en-3-ol) are likely to be from the decomposition of a terephthalate.

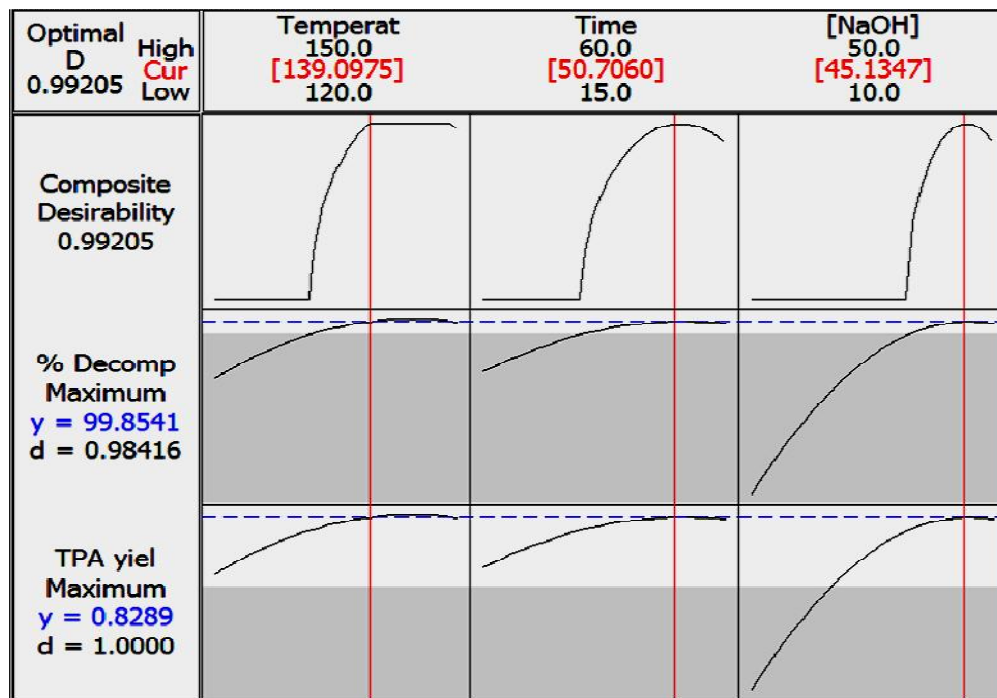


Fig. 8. Optimization plots for the alkaline solvolysis of PET for the conditions under study

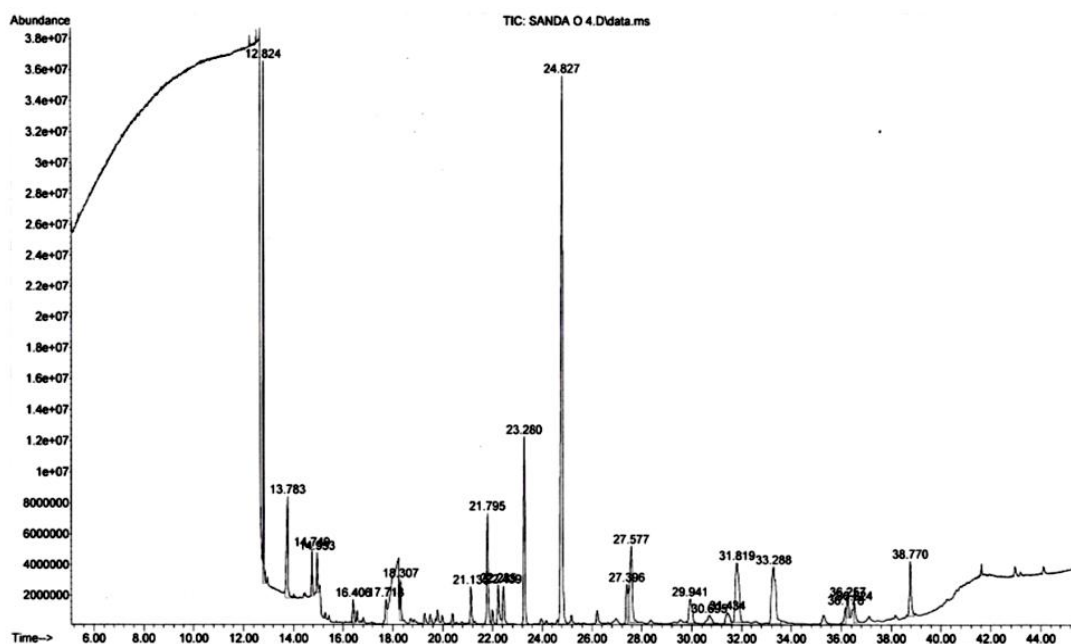


Fig. 9. Gas chromatograph for the filtrate obtained from the alkaline solvolysis of PET in butan-1-ol media

Table 6. Some of the identified components in the filtrate

Peak no	Identified component	RT (min)	Peak Area%
1	2-Methyl-1-butanol	12.824	8.16
2	Butyl acetate	13.783	4.74
3	2-Butoxyethanol	14.749	1.29
4	2-Ethyl-1-hexanol	17.718	1.41
5	(E)-Nonen-1-ol	18.307	1.56
6	1-Butoxy-1-isobutoxy-butane	21.795	4.96
8	2-Oxooctanoic acid	22.439	1.85
7	1,1-Diisobutoxy-isobutane	23.280	7.67
9	1,1-Dibutoxy-butane	24.827	30.44
10	2-Hydroxy-tetradecanoic acid	27.396	2.01
12	Butoxy-2,4-dimethyl-1-pentene	29.941	2.53
11	2-Ethylnon-1-en-3-ol	31.819	6.90

4. CONCLUSION

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

reactions, implying that postconsumer PET can be a good source of terephthalic acid, ethylene glycol and other value-added products.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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