Effect of Flake Pigmentation on the Microwave-Assisted Alkaline Solvolysis of Postconsumer Polyethylene Terephthalate in Primary $C_1 – C_3$ Aliphatic Alcohols

O. Sanda[1*], E. O. Ehinmitola[1], A. Y. Kolapo[1] and E. A. Taiwo[1]

[1]*Department of Chemical Engineering, Obafemi Awolowo University, Ile-Ife, Nigeria.*

Authors' contributions

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

Article Information

Original Research Article

ABSTRACT

This study was carried out to examine the alkaline solvolysis of pigmented postconsumer polyethylene terephthalate (PET) in solutions of $C_1 – C_3$ primary alcohols via microwave heating. The effect of various process parameters such as flake pigmentation, time and sodium hydroxide concentration on the degree of PET degradation and product yield were studied for each alcohol. Response surface methodology (RSM) was used for predicting the optimal conditions for alkaline solvolysis of PET scrap, with Central Composite Design (CCD) for the two parameters chosen as the experimental design. The data obtained from measurement of properties were fitted as second-order equations. The findings of this study showed that the yield is independent of the pigmentation and that microwave-assisted alkaline solvolysis of pigmented postconsumer PET resulted in higher conversion within a shorter processing time, compared to conventional heating methods with identical products obtained in each case.

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Corresponding author: E-mail: osanda@ouaife.edu.ng, sanchemie02@gmail.com, osanda@oauife.edu.ng;

1. INTRODUCTION

Polyethylene terephthalate (PET) is a thermoplastic polymer of choice in the production of bottles for carbonated drinks due to its glass-like transparency coupled to gas barrier properties for the retention of carbonation. In addition, PET exhibits a high toughness/weight property ratio, which allows for the production of lightweight and securely unbreakable containers with large capacity [1]. PET is also used in other industries such as textiles [2] the production of building and construction materials [3-6], and polyester-based adhesives and coatings [7-15]. However, disposing postconsumer PET bottles using conventional methods such as landfills and incineration is a major problem because of the non-biodegradability of PET. Although recycling is considered to be one of the approaches to solve the accumulation of PET waste, however PET bottles are available in different colours (such as transparent, blue, green or brown). Unfortunately, only transparent (or lightly-tinted blue) PET bottles are high valued in today's recycling market, as coloured PET bottles have limitations for their reuse and therefore have a much lower market value [16].

Taking into account the cost of processing the so-called "problematic" coloured PET bottles, an alkaline solvolysis approach was chosen for this study because it operates under less hazardous conditions, eliminating the need for corrosion resistant pressure vessels [17-24]. The aim of this study was to explore the application of microwave heating during the processing of pigmented PET via an alkaline solvolysis route with a view to examine the effects of various parameters such as pigmentation, reaction time and alkali dosage on the process and compare the results with that obtained from conventional heating methods reported in an earlier work.

2. EXPERIMENTAL

2.1 Materials

Postconsumer PET bottles of different pigmentations (transparent, blue, brown) were sourced from restaurants, fast foods outlets and hotels in Ile-Ife, Nigeria. Methanol (>98.5% w/w), ethanol and hydrochloric acid (37% w/w) were supplied by BDH Chemicals (Poole, England), propan–1–ol (> 99% w/w) was supplied by Loba Chemie (Mumbai, India), sodium hydroxide was supplied by J T Baker (Philipsburg, USA) and pyridine by Merck, Millipore (Darmstadt, Germany). All chemicals are of analytical grade and were used as-received. The collected PET bottles were shredded to small sizes of approximately 5×5 mm, 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 in Alcoholic Media

About 5 g of PET flakes and 100 mL of sodium hydroxide solution in methanol were charged into a 250 mL round-bottomed flask which was fitted with a reflux condenser. Heating was by means of a microwave oven (microwave power, 700 W; frequency, 2.43 GHz) as shown in Fig. 1, with each experiment designed according to a three-level, two-factor Central Composite Design (CCD), which generated 13 experiments per alcohol. The parameters investigated were reaction time (x_1) and sodium hydroxide concentration (x_2), with experimental design matrix, and combination of parameters for each experiment as presented in Tables 1 and 2, respectively. The response for each alcohol was evaluated using Minitab statistical software (version 16.1.1) and fitted to the quadratic model below:

$$Y = \delta_o + \delta_1 X_1 + \delta_2 X_2 + \delta_{12} X_1 X_2 + \delta_{11} X_1^2 + \delta_{22} X_2^2 \tag{3}$$

Where Y is the predicted response (% PET decomposition), δ_o is the intercept term, δ_1, δ_2 are the linear coefficients, δ_{12} is the interaction term, and δ_{11}, δ_{22} are the quadratic coefficients.

In addition, the terms X_1 and X_2 are coded factors which are related to the actual factors x_1 and x_2 by:

$$X_i = \frac{x_i - x_o}{\Delta x} \tag{4}$$

where *Xi* is the coded value for the ith input (that is x_i), *x*o is 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.

At the end of each experiment, 100 mL of distilled water was added to the mixture and stirred until the depolymerisation products dissolved. The mixture was filtered using an ashless filter paper to remove the unreacted PET which was washed with distilled water, dried at 105 ^{o}C and weighed. To the filtrate, 100 mL of 1 M HCl was added dropwise with constant stirring and the white precipitate which appeared was separated by filtration, washed with distilled water to remove water soluble impurities, filtered again, dried at 105°C and weighed. The entire process was repeated for ethanol and propan-1-ol.

The percentage decomposition of PET was determined by gravimetry using the formula:

$$\% \text{ Decomposition of PET} = \left(\frac{W_o - W_f}{W_o}\right) \times 100 \quad (2)$$

where W_o is the initial mass of PET flakes and W_f is the mass of unreacted PET at the end of each experiment.
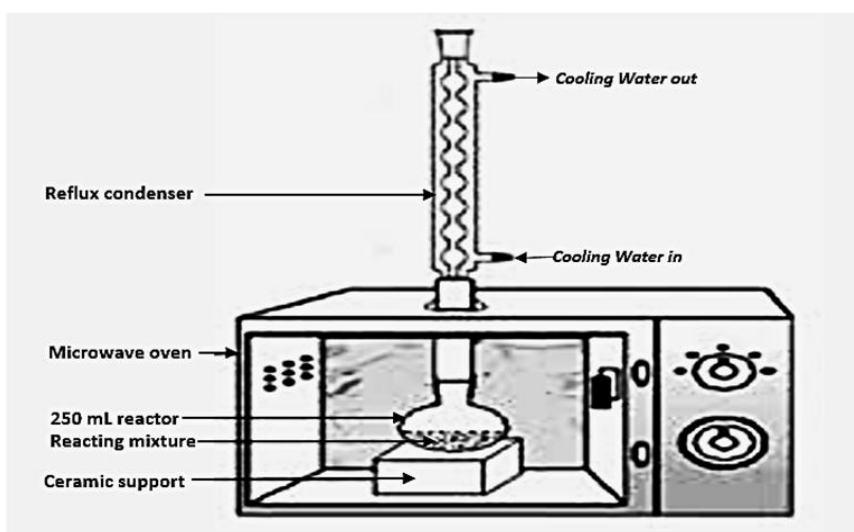

Fig. 1. A schematic representation of the experimental setup used in this study

Table 1. Central composite design for the microwave-assisted PET decomposition process

Factors	Coded levels				
	− α	− 1	0	+ 1	+ α
Time (min)	7.93	10.00	15.00	20.00	22.07
NaOH concentration (g/L)	25.86	30.00	40.00	50.00	54.14

Table 2. Combination of process parameters for each experiment

Experiment no.	Time (x_1), min.	NaOH concentration in alcohol (x_2), g/L
1.	15	25.86
2.	15	40
3.	7.93	40
4.	15	54.14
5.	22.07	40
6.	15	40
7.	10	50
8.	10	30
9.	15	40
10.	20	30
11.	15	40
12.	15	25.86
13.	15	40

2.3 Acid Value Determination

1 g of the product obtained from each of the alkaline solvolysis experiments was accurately weighed into a 100 mL beaker, containing 25 mL of pyridine. The mixture was stirred till the suspension was completely homogenous, after which 25 mL of distilled water and 2 – 3 drops of phenolphthalein indicator were added. The solution was titrated against 0.5 M potassium hydroxide solution till a permanent pink end point was obtained. A blank determination was also carried out. The acid value was determined from the following formula [25]:

$$AV \, (mg \, KOH/g) \, = \, \frac{56.1 \times M \times (V_S - V_B)}{w} \qquad (1)$$

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

2.3 Fourier Transform Infra-red (FTIR) Spectroscopy

FTIR spectroscopy was done on the white precipitates obtained from the decomposition of each PET type to determine the nature of functional groups present in each product. Infrared spectroscopy was carried out on a Thermo Nicolet iS5 FT-IR equipped with iD3 Attenuated Total Reflectance (ATR) assessor and Omnic FTIR software for spectra processing and analysis.

3. RESULTS AND DISCUSSION

3.1 Comparison between Conventional and Microwave Heating for Alkaline Solvolysis of PET

The decomposition of PET flakes in a 40 g/L solutions of sodium hydroxide in the alcohols was done by heating the reacting mixture under reflux using a 1500 W heating mantle (i.e. a conventional heating approach) for 20 min. The process was repeated using a 700 W microwave oven and the results are presented in Figs. 2 – 4. The microwave-assisted alkaline solvolysis of PET resulted in a higher conversion of PET, compared to conventional heating, irrespective of pigmentation. For ethanol and propan-1-ol media, the microwave-assisted reactions gave a higher conversion, although the general trend is not different from that reported in a previous work [24]. Based on the theory of PET alkaline solvolysis, the expected primary product is terephthalic acid [24,26]. For each experiment, a white powder was obtained and was subsequently subjected to various physical and chemical tests.

3.1.1 Acid value

The acid values of the solid products obtained from the alkaline solvolysis runs in methanol media gave an average of 673.4, 671.7 and 671.3 mg KOH/g for clear, blue and brown PET, respectively (Table 3). For the ethanol media, average acid values of 671.7, 667.3 and 663.9 mg KOH/g for clear, blue and brown PET, respectively were evident. Propan-1-ol media average values were 665.8, 663.9 and 661.4 mg KOH/g for clear, blue and brown. Comparing these values with the theoretical acid value of TPA (i.e. 675 mg KOH/g), it can be deduced that the observed variations might have been a result of impurities/ additives present in the PET used.

3.1.2 FTIR spectroscopy analysis

The FTIR spectra of the products obtained from the microwave-assisted decomposition of PET using 40 g/L NaOH in the alcohols are shown in Figs. 5 – 7. The infra-red (IR) spectra indicated that the products obtained are identical, irrespective of the PET pigmentation or the alcohol used. Considering the absorption bands at 1600 and 1400 cm^{-1} (–C-C– stretch for aromatic compounds) and the sharp absorption band around 750 cm^{-1}, the products can be *p*-substituted aromatic compounds. The broad –OH band occurring between 3000 and 3500 cm^{-1}, the –C=O band around 1700 cm^{-1} and the –C–O

Table 3. Acid values for the aliphatic alcohols

PET flake pigmentation	Alcohol used		
	Methanol	Ethanol	Propan–1–ol
Clear PET	673.4	671.7	671.3
Blue PET	668.1	667.3	668.8
Brown PET	665.8	663.9	669.4

bands around 1300 cm^{-1} indicate that the products are carboxylic acids. These bands occur around 1280 cm^{-1} on the figure inserted below (as indicated by the red circled bands in Fig. 5).
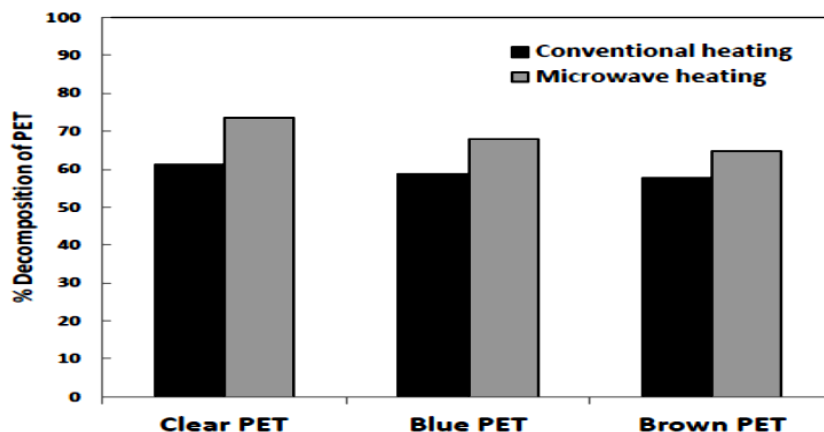

Fig. 2. Comparison between conventional and microwave-assisted heating of PET decomposition in a methanol media
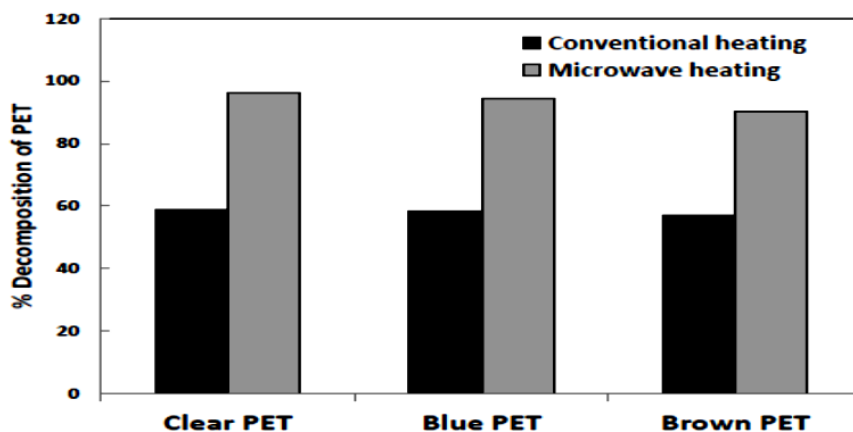

Fig. 3. Comparison between conventional and microwave-assisted heating of PET decomposition in an ethanol media
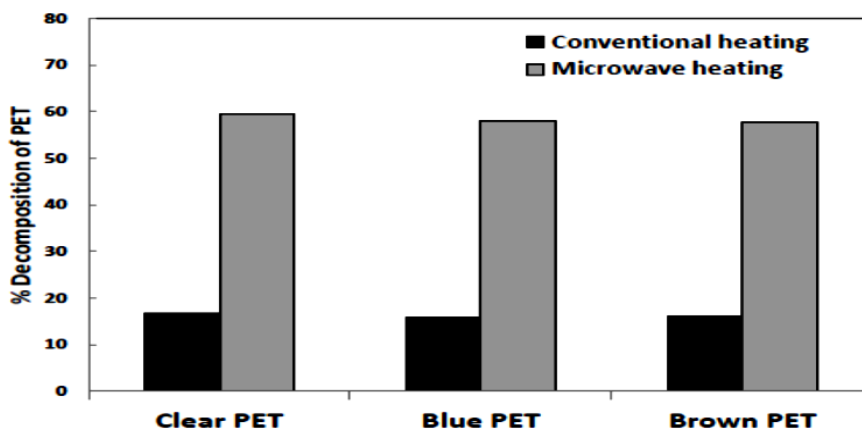

Fig. 4. Comparison between conventional and microwave-assisted heating of PET decomposition in a propan-1-ol media

3.2 PET Solvolytic Decomposition Study

Studies done so far on the alkaline solvolysis of postconsumer PET in alcoholic media showed that it is possible to decompose PET according to the reaction mechanisms shown below [24,26]:

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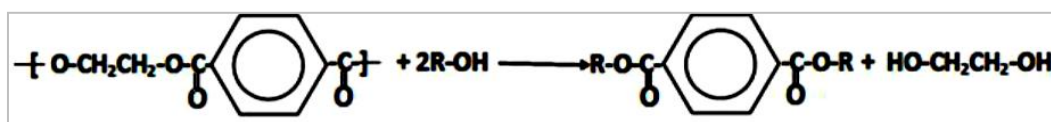

Saponification step: The dialkyl terephthalate from the alcoholysis step reacts with an alkali such as sodium hydroxide (NaOH) to yield the corresponding salt:
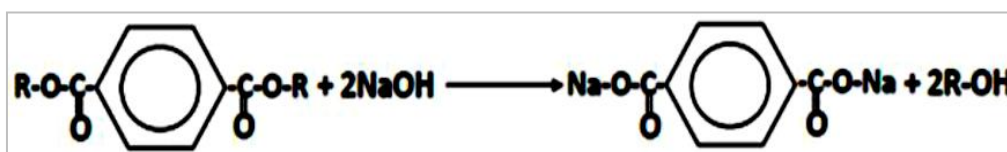
The sodium salt formed in the saponification step can be broken down to yield terephthalic acid and the corresponding salt of the alkali used using a mineral acid:
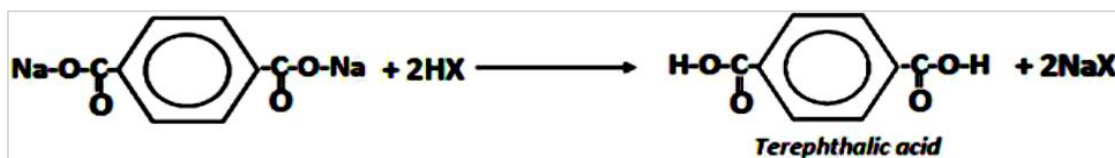
Where HX represents HCl or HNO_3. From the reaction steps shown above, the alcohol serves as "solvent", as it is recovered in the saponification stage. The overall chemical equation for the alkaline solvolysis of PET in alcoholic media to yield disodium terephthalate (Na_2TPA) and ethylene glycol (EG) may be written as shown:

$$PET + 2NaOH \xrightarrow{alcohol} Na_2TPA + EG$$
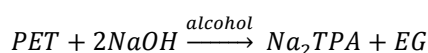
The extent of conversion of PET in the equations presented above depends on the molecular mass of the alcohol used and its boiling point. The extent of conversion tends to decrease with molecular mass of the alcohol used as reported by Sanda et al. [24]. However, the trend observed in Figs. 2-4 suggests an initial lag in conversion rate for the methanol medium due to the formation of a layer of intermediate products on the flakes, according to the kinetic model reported in an earlier work for the alkaline solvolysis of PET using NaOH in butanol media [26].

The relationship between the response (% decomposition of PET) and the independent variables (reaction time and alkali concentration) were studied for the various alcohols and PET type in order to optimize the alkaline solvolysis of PET. The coefficients of the final model equations in terms of the coded factors are given in Tables 4 – 6 for PET decomposition, while the results of the analysis of variance (ANOVA) for the response surface models are shown in Tables 7 – 9. The quadratic models are significant ($p < 0.05$), accounting for over 96 % of the observations. The extent of PET decomposition depends on the reaction time and the concentration of NaOH for the alcohols studied. The results obtained follow similar trends, irrespective of the PET pigmentation.

The response surface contour plots of PET decomposition in relation to sodium hydroxide concentration and reaction time are illustrated in Figs. 8 – 10. Sodium hydroxide concentration and reaction times were kept at mid-point levels for each pigment type, where the labels (a), (b) and (c) represent clear, blue and brown PET, respectively. From the studies, it was found that

irrespective of the PET pigmentation, the reaction time and alkali concentration play an important role in the alkaline solvolysis of PET in alcoholic media. It was also found that for the pigmented PET flakes, the colouring matter was soluble in the alcohol used, suggesting that the crude product can be further purified by washing it with the alcohol used in the process.
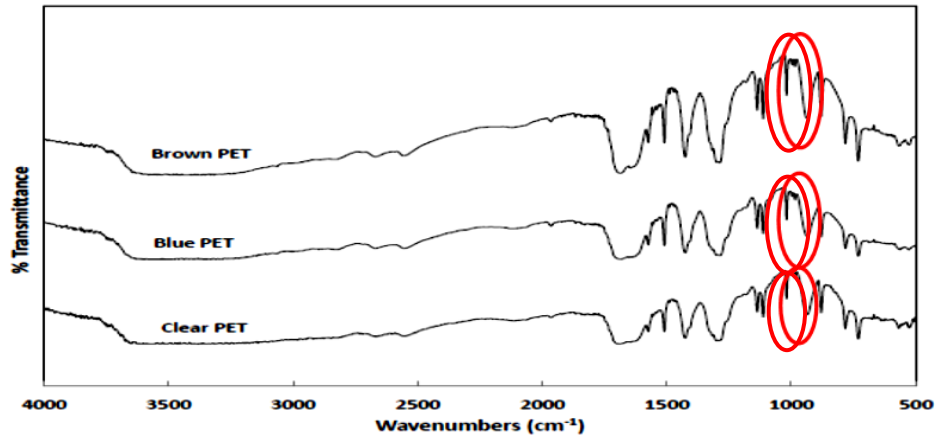

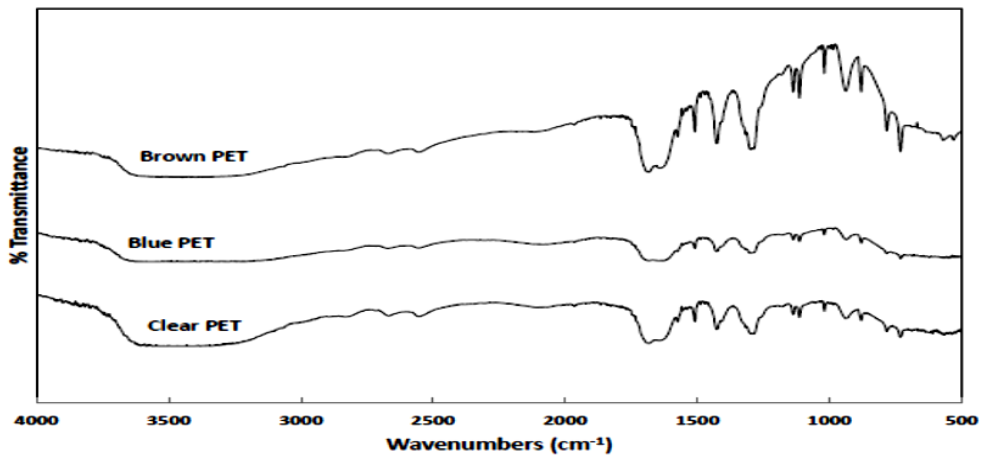**Fig. 5. IR spectra for the alkaline solvolysis products obtained from methanol media**

Fig. 6. IR spectrum of the alkaline solvolysis products obtained from ethanol media
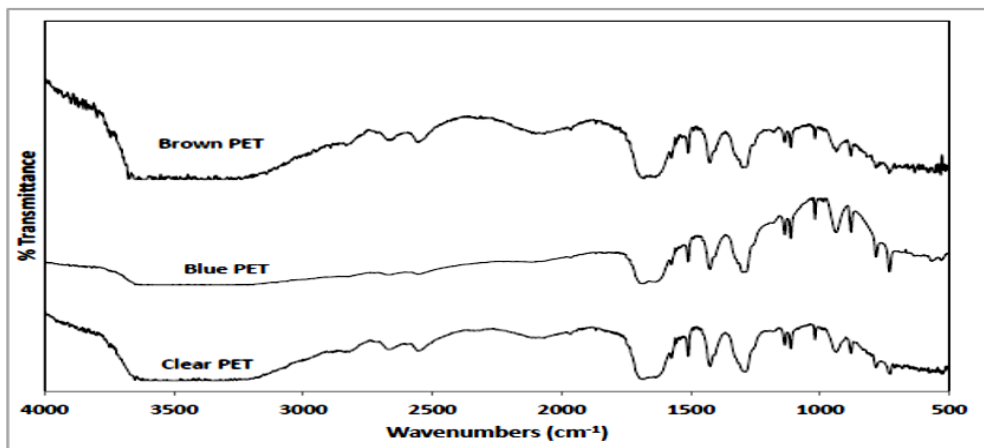

Fig. 7. IR spectrum of the alkaline solvolysis products obtained from propan-1-ol media

Table 4. Regression coefficients of fitted equations for the percentage decomposition for clear PET ($Y_{clear} = \delta_o + \delta_1 X_1 + \delta_2 X_2 + \delta_{12} X_1 X_2 + \delta_{11} X_1^2 + \delta_{22} X_2^2$)

Coefficients	Methanol	Ethanol	Propan-1-ol
δ_o	53.125	63.225	39.3709
δ_1	11.738	18.091	10.7324
δ_2	2.919	11.778	6.5919
δ_{12}	2.280	4.431	4.9755
δ_{11}	-3.865	1.008	1.8363
δ_{22}	-1.475	-2.250	0.4410
R^2	0.9922	0.9985	0.9776
Adjusted R^2	0.9862	0.9974	0.9616

Table 5. Regression coefficients of fitted equations for the percentage decomposition for blue PET ($Y_{blue} = \delta_o + \delta_1 X_1 + \delta_2 X_2 + \delta_{12} X_1 X_2 + \delta_{11} X_1^2 + \delta_{22} X_2^2$)

Coefficients	Methanol	Ethanol	Propan-1-ol
δ_o	53.176	62.8201	40.2046
δ_1	12.771	17.8127	10.4114
δ_2	2.927	11.8245	6.7474
δ_{12}	2.711	4.0553	4.4305
δ_{11}	-3.984	0.8763	1.7126
δ_{22}	-1.800	-2.1600	0.4500
R^2	0.9866	0.9978	0.9942
Adjusted R^2	0.9771	0.9963	0.9900

Table 6. Regression coefficients of fitted equations for the percentage decomposition for brown PET ($Y_{brown} = \delta_o + \delta_1 X_1 + \delta_2 X_2 + \delta_{12} X_1 X_2 + \delta_{11} X_1^2 + \delta_{22} X_2^2$)

Coefficients	Methanol	Ethanol	Propan-1-ol
δ_o	53.671	61.0857	38.8460
δ_1	12.385	15.9010	9.8524
δ_2	2.944	10.8372	6.7406
δ_{12}	2.289	3.0275	4.4535
δ_{11}	-3.993	0.1378	1.2490
δ_{22}	-2.295	-3.2900	0.5635
R^2	0.9791	0.9950	0.9897
Adjusted R^2	0.9641	0.9915	0.9824

Table 7. ANOVA for the response model for the % decomposition of the clear PET

Source	Degrees of freedom	Sum of squares	Mean square	F-value	P-value
PET solvolysis in methanol media					
Model	5	1444.29	288.86	177.27	<0.001
Residuals	7	11.41	1.63		
Lack of fit	3	7.90	2.63	3.00	0.158
Pure error	4	3.51	0.88		
PET solvolysis in ethanol media					
Model	5	4181.45	836.29	906.40	<0.001
Residual	7	6.46	0.92		
Lack of fit	3	6.44	2.15	502.26	<0.001
Pure error	4	0.02	0.00		
PET solvolysis in propan-1-ol media					
Model	5	1528.63	305.726	61.11	<0.001
Residual	7	35.02	5.003		
Lack of fit	3	35.02	11.672	12703.98	<0.001
Pure error	4	0.00	0.001		

Table 8. ANOVA for the response model for the % decomposition of the blue PET

Source	Degrees of freedom	Sum of squares	Mean square	F-value	P value
PET solvolysis in methanol media					
Model	5	1686.55	337.31	103.21	<0.001
Residuals	7	22.88	3.27		
Lack of fit	3	15.73	5.24	2.93	0.163
Pure error	4	7.15	1.79		
PET solvolysis in ethanol media					
Model	5	4087.83	817.57	645.16	<0.001
Residual	7	8.87	1.27		
Lack of fit	3	8.55	2.85	35.58	0.002
Pure error	4	0.32	0.08		
PET solvolysis in propan-1-ol media					
Model	5	1458.40	291.681	239.08	<0.001
Residual	7	8.54	1.220		
Lack of fit	3	7.24	2.413	7.42	0.041
Pure error	4	1.30	0.325		

Table 9. ANOVA for the response model for the % decomposition of the brown PET

Source	Degrees of freedom	Sum of squares	Mean square	F-value	P value
PET solvolysis in methanol media					
Model	5	1594.85	318.97	65.45	<0.001
Residuals	7	34.11	4.87		
Lack of fit	3	20.51	6.84	2.01	0.255
Pure error	4	13.60	3.40		
PET solvolysis in ethanol media					
Model	5	3337.01	667.40	279.67	<0.001
Residual	7	16.70	2.39		
Lack of fit	3	16.24	5.41	46.64	0.001
Pure error	4	0.46	0.12		
PET solvolysis in propan-1-ol media					
Model	5	1377.26	275.451	134.61	<0.001
Residual	7	14.32	2.046		
Lack of fit	3	12.96	4.320	12.65	0.016
Pure error	4	1.37	0.341		

Table 10. Typical vibrational frequencies for selected functional groups

Bond	Molecule	Wavenumber (cm^{-1})
C–O	Alcohols, ethers, esters, carboxylic acids, etc.	1300 – 1000
C=O	Aldehydes, ketones, esters, carboxylic acids	1750 – 1680
C=O	Amides	1680 – 1630
N–H (Stretching)	Amines and amides	3500 – 3100
–N–H (Bending)	Amines and amides	1640 – 1550
O–H	Alcohols	3650 – 3200
C–N	Amines	1350 – 1000
S–H	Mercaptans	2550

(Obtained from https://nptel.ac.in/courses/102103044/module2/lec10/6.html)
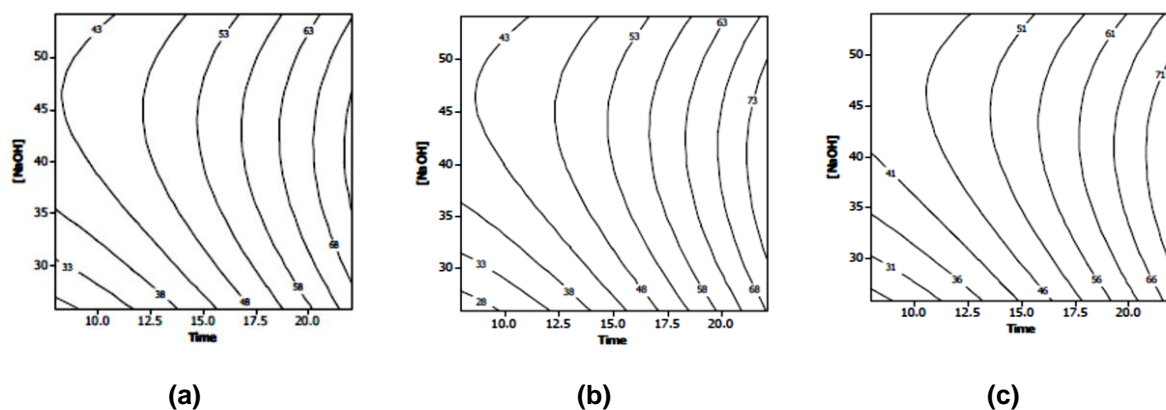
Fig. 8. Contour plots for the microwave-assisted decomposition of PET in methanol media
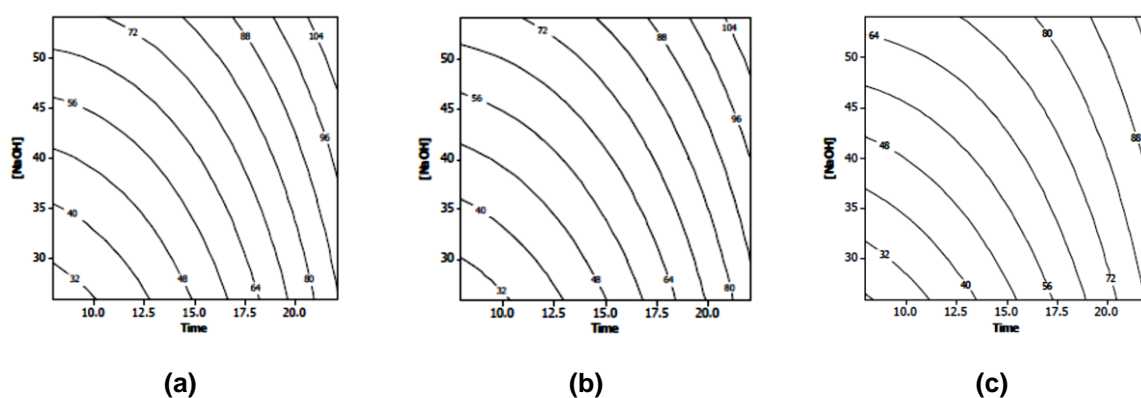

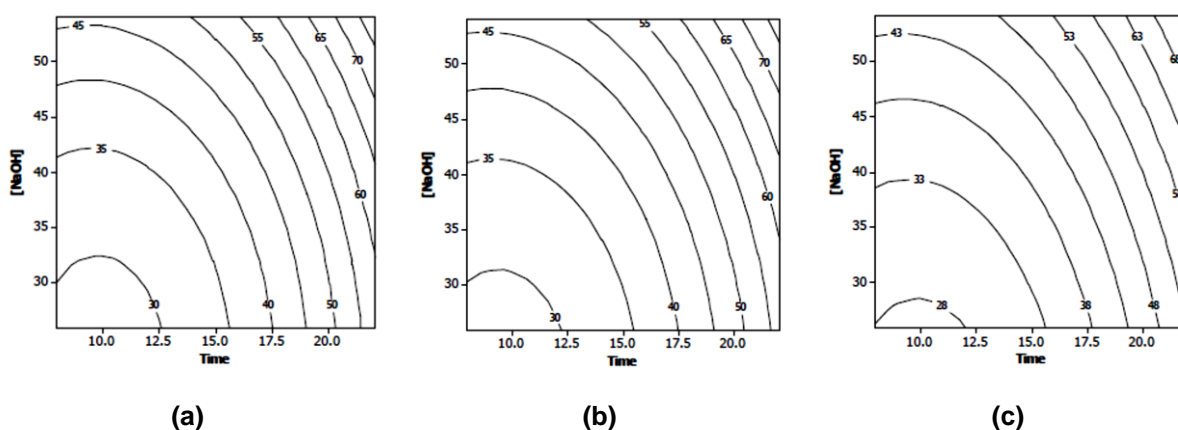**Fig. 9. Contour plots for the microwave-assisted decomposition of PET in ethanol media**

Fig. 10. Contour plots for the microwave-assisted decomposition of PET in propan-1-ol media

4. CONCLUSION

This study has shown that irrespective of the pigmentation of the PET flakes, microwave-assisted alkaline solvolysis has proven to be a simple and effective method for processing postconsumer PET into value added products. Irrespective of the alcohol used, the rate of decomposition of PET only depends on the concentration of alkali and process time, yielding identical products in each case. Additionally, this method does not require adverse processing conditions thereby resulting in higher conversions within a short period time (typically less than 30 min), compared to processing methods requiring conventional heating methods.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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