Original Research Article

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

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~~s~~ yield were studied for each alcohol. Response surface methodology (RSM) was used for predicting the optimal conditions for ~~the~~ alkaline solvolysis of PET scrap, with Central Composite Design (CCD) for the two variables 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 ~~gives~~ higher conversion within a shorter processing time, compared to conventional heating methods with identical products obtained in each case.

1.0 INTRODUCTION

Polyethylene terephthalate (PET) is a thermoplastic polymer of choice in the production of beverage bottles due to its glass-like transparency coupled to ~~with adequate~~ 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 ~~has~~ also ~~found~~ used in ~~various rather~~ ~~unconventional~~ other industries ~~areas~~ such as ~~clothing and~~ textiles [2] ~~in~~ the production of building and construction materials [3–6], and polyester-based adhesives and coatings [7 – 15].

However, ~~the non-biodegradability of PET is the major obstacle to~~ disposing postconsumer PET bottles using conventional methods such as landfill~~ings~~ and incineration is a major problem because of the non-biodegradability of PET. Although recycling is considered ~~assumed~~ to be one of the ~~best~~ approaches to solve the accumulation of PET waste ~~problem~~, however ~~there is need consider the fact that~~ PET bottles are available ~~come~~ in different ~~various~~ colours (such as transparent~~clear~~, blue, green or brown). Unfortunately, only transparent ~~colourless~~ (or lightly-tinted blue) PET bottles ~~offer reclaimers~~ ~~a~~are high

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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, ~~effectively~~ eliminating the need for corrosion resistant pressure vessels [17 – 24]. The aim of this study was~~is~~ to explore the ~~applicability~~ application of microwave heating during ~~on~~ the processing of pigmented PET via an alkaline solvolysis route with a view to examine ~~ing~~ 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.~~ 2.0 Experimental

2.1. Materials

Postconsumer PET bottles of different pigmentations (transparent~~clear~~, green, brown) were sourced from restaurants, fast foods outlets and hotels in Ile-Ife, Nigeria. Methanol ~~(BDH),~~ and ethanol were supplied by ~~(~~BDH (Where?), propan–1–ol supplied by Loba Chemie (Where?)~~(Lobachemie)~~, sodium hydroxide supplied by ~~(~~J~~.~~ T~~.~~ Baker (Where?) and pyridine by ~~(~~Merck, Millipore (Where? USA? Which city? etc) ~~were obtained from reputable chemical stores.~~ All reagents were used as-received. The collected PET bottles were crushed ~~shredded~~, washed and dried in an oven at 110 ^{o}C for 4 h and after drying, stored in airtight plastic containers prior use. Please include the quality/grade of the reagents used.

2.2 Alkaline Solvolysis of PET in Alcoholic Media

About 5 g of PET flakes and 100 mL~~l~~ of ~~a solution of~~ sodium hydroxide in methanol were charged into a 250 mL~~l~~ round-bottomed flask which was fitted with a reflux condenser. Heating was by means of ~~using~~ a microwave oven (microwave power, 700 W; frequency, 2.43 GHz) as shown in ~~-~~Figure 1. At the end of each run, 100 mL~~l~~ of water was added to the ~~and the~~ mixture and stirred until the depolymerisation products dissolved. The mixture was filtered using an ashless filter paper, and the filtrate obtained was washed with distilled water, dried at 105 ^{o}C and weighed. 1 M HCl was added dropwise to the filtrate ~~obtained~~ with constant stirring. T~~and t~~he white precipitate which appeared was separated by filtration, washed with distilled water to remove water soluble impurities, filtered again, dried at 105 ^{o}C and weighed. The entire process was repeated for ethanol and propan-1-ol.

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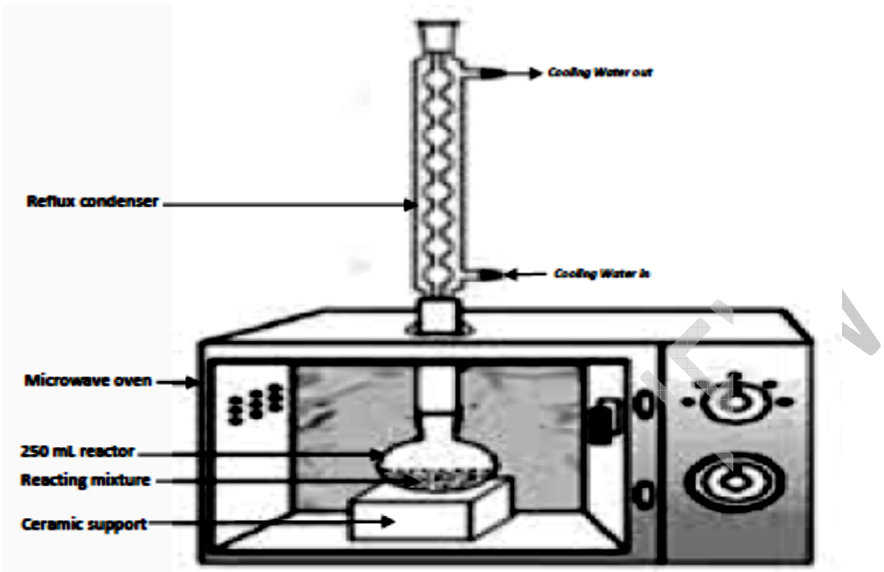

Figure 1: A schematic representation of the experimental setup used in this study

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

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

where W_o is the initial mass of PET flakes and W_f is the mass of unreacted PET at the end of each run. In order to optimize the alkaline solvolysis process, a three-level-two factor Central Composite Design (CCD) was employed, with 13 experimental runs per alcohol. The parameters ~~factors~~ investigated were reaction time (x_1) and alkali concentration (x_2). The response for each alcohol was evaluated using Minitab statistical software (version 16.1.1) and fitted to the quadratic model below:

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

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

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

where Xi is the coded value for the i^{th} input (that is~~,~~ x_i), xo 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. The experimental design matrix for PET decomposition in methanol and ethanol media is presented in Table 1.

Table 1: Central Composite Design for the Microwave-assisted PET decomposition process

	Coded levels				
Factors	**– 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

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2.3. Acid value determination

~~About~~ 1 g of the product~~s~~ obtained from each of the alkaline solvolysis runs was accurately weighed into a 100 mL~~l~~ beaker, containing 25 mL~~l~~ of pyridine. The mixture was stirred till the suspension ~~sample~~ was completely homogenous ~~dissolved~~, after which 25 ~~ml~~ mL of 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~~, excluding the sample~~. The acid value was determined from the following formula [25]:

$$\text{AV (mg KOH/g)} = \frac{56.1 \times M \times (V_s - V_B)}{w} \quad (1)$$

w~~W~~here 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.

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3. Results and Discussion

3.1. Comparison between~~of~~ conventional and microwave heating for ~~the~~ alkaline solvolysis of PET

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

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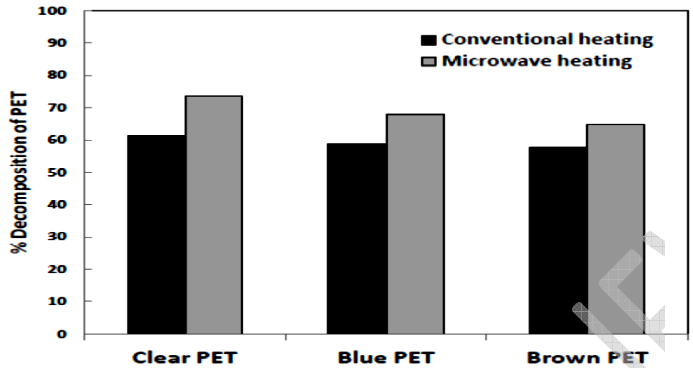

Figure 2: Comparison between conventional and microwave-assisted heating of PET decomposition in a methanol media.~~ for conventional and microwave-assisted heating~~

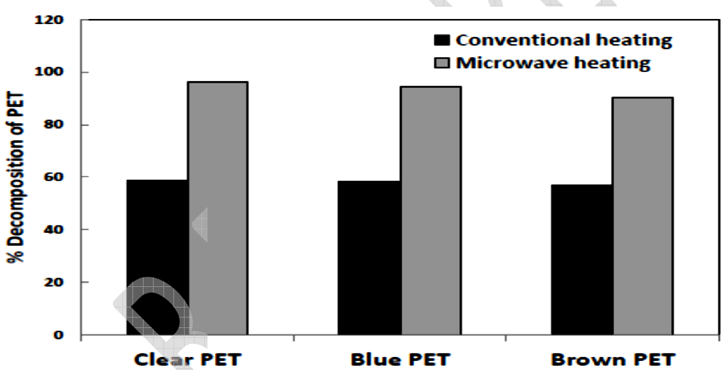

Figure 3: Comparison between conventional and microwave-assisted heating of PET decomposition in an ethanol media.~~ for conventional and microwave-assisted heating~~

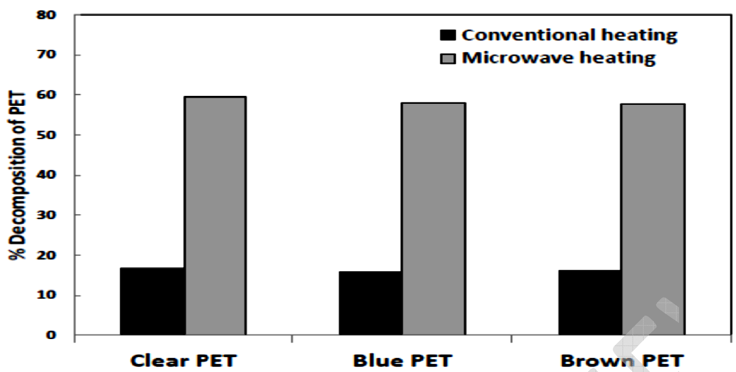

Figure 4: Comparison between conventional and microwave-assisted heating of PET decomposition in a propan-1-ol media ~~for conventional and microwave-assisted heating~~.

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 transparent~~clear~~, blue and brown PET, respectively (Table 2). For the ethanol media, average acid values of 671.7, 667.3 and 663.9 mg KOH/g for transparent~~clear~~, blue and brown PET, respectively were evident. P~~For the p~~ropan-1-ol media ~~, the~~ average values were ~~of~~ 665.8, 663.9 and 661.4 mgKOH/g for transparent, blue and brown.~~the propan-1-ol media as shown in Table 2.~~ Comparing these values with the theoretical acid value~~number~~ of TPA (~~that is,~~i.e. 675mg KOH/g), it can be deduced ~~said~~ that the observed variations might have been a result of impurities/ additives present in the PET used.

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3.1.2. Fourier-transport infra-red spectroscopy ~~TIR~~ analysis

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The FTIR spectra of the products obtained from the microwave-assisted decomposition of PET using 40 g/L~~l~~ NaOH in the alcohols are shown in Figures 5 – 7. The ~~A look at 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 ~~said to be~~ *p*-substituted aromatic compounds. The ~~very~~ broad –OH band occurring between 3000 and 3500 cm^{-1}, and the –C=O band around 1700 cm^{-1} indicate that the products are carboxylic acids.

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Table 2: Acid Values for the Aliphatic Alcohols

PET flake pigmentation	Alcohol used

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	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

3.2. PET s~~S~~olvolytic d~~D~~ecomposition s~~s~~tudy. ~~u~~~~Using~~ ~~r~~~~Response~~ ~~s~~~~Surface~~ ~~m~~~~Methodology~~

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 3 – 5 for PET decomposition, while the results of the ~~ANOVA~~ analysis of variance (ANOVA) ~~analyses~~ for the response surface models are shown in Tables 6 – 8. The quadratic models are significant (~~*P*~~ *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. T~~, and t~~he results obtained follow similar trends, irrespective of the PET pigmentation.

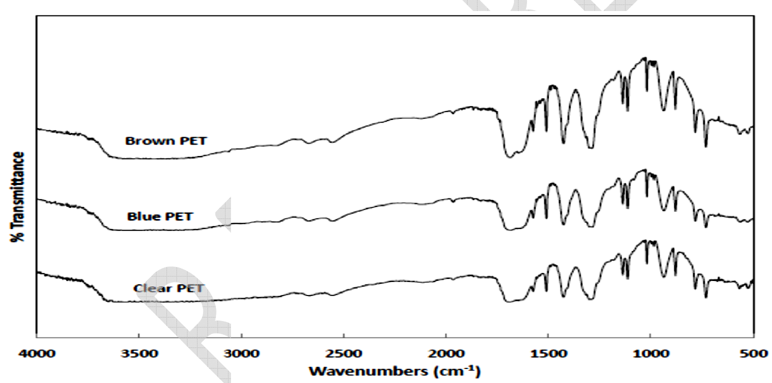

Figure 5: IR spectra for the alkaline solvolysis products obtained from methanol media

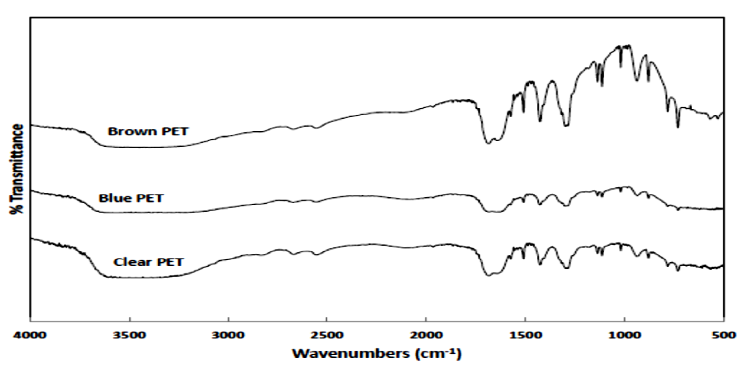

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Figure 6: IR spectrum of the alkaline solvolysis products obtained from ethanol media

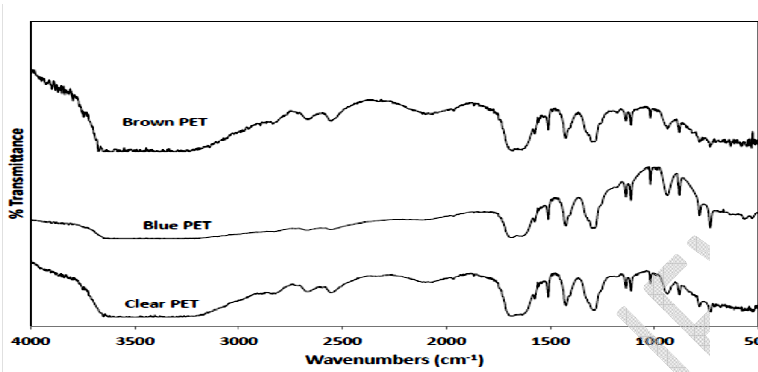

Figure 7: IR spectrum of the alkaline solvolysis products obtained from propan-1-ol media

Table 3: Regression cCoefficients 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	~~**Alcohol used**~~		
	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 4: Regression cCoefficients 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	~~**Alcohol used**~~		
	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

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R^2	0.9866	0.9978	0.9942
Adjusted R^2	0.9771	0.9963	0.9900

Table 5: Regression c~~C~~oefficients of fitted equations for the percentage decomposition for brown PET

$$(Y_{brown} = \delta_o + \delta_1 X_1 + \delta_2 X_2 + \delta_{12} X_1 X_2 + \delta_{11} X_1^2 + \delta_{22} X_2^2)$$

	~~**Alcohol used**~~		
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 6: ANOVA for the response model for the % decomposition of the clear PET

Source	**DF**	**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		

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Table 7: ANOVA for the Response Model for the % decomposition of the blue PET

PET solvolysis in methanol media

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Source	DF	Sum of Squares	Mean Square	F-value	P value
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 8: ANOVA for the Response Model for the % decomposition of the brown PET

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Source	DF	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		

The response surface contour plots of PET decomposition in relation to sodium hydroxide concentration and reaction time are illustrated in Figures 8 – 10. S~~, with s~~odium hydroxide concentration and reaction times were kept at ~~their~~ 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~~observed~~ that for the pigmented PET flakes, the colouring matter was~~is~~ soluble in the alcohol used, suggesting that the crude product can be further purified by washing it with the alcohol used in the process.

4. Conclusion

This study has shown that irrespective of the pigmentation of the ~~postconsumer~~ PET flakes, microwave-assisted alkaline solvolysis has proven~~d~~ 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, t~~, since t~~his method does not require ~~is devoid of the need for~~ adverse processing conditions thereby ~~and~~ resulting in~~gives~~ higher conversions within a short period time, typically less than 30 min, compared to conventional heating methods.

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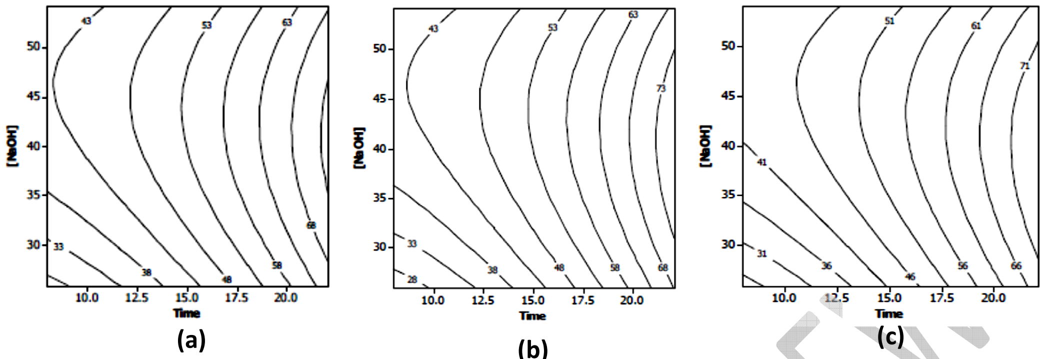

Figure 8: Contour plots for the microwave-assisted decomposition of PET in methanol media

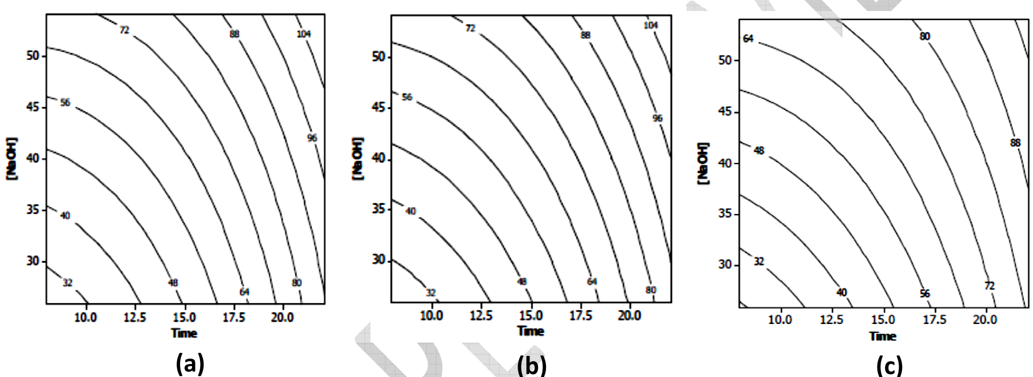

Figure 9: Contour plots for the microwave-assisted decomposition of PET in ethanol media

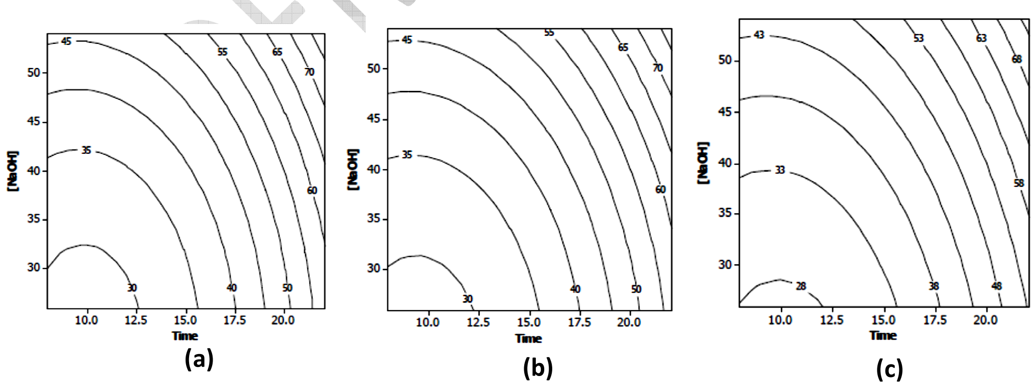

Figure 10: Contour plots for the microwave-assisted decomposition of PET in propan-1-ol media

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Comment [PM28]: This hyphen is not the same as the hyphens that follow.

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