HYDROLYSIS OF POLY-ETHYLENE TEREPHTHALATE WASTE USING HIGH PRESSURE AUTOCLAVE: A CHEMICAL RECYCLING

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ABSTRACT

High pressure autoclave (HPA) is used for high-pressure and high-temperature chemical reactions like hydrolysis, alkylation, amination, bromination, carboxylation, catalytic reduction, chlorination, dehydrogenation, esterification, ethoxylation, halogenation, hydrogenation, methylation, nitration, oxidation, ozonization, polymerization, sulphonation. Chemical recycling by hydrolysis of Polyethylene Terephthalate (PET) waste was studied at different temperatures and autogeniuos pressures were recorded. Temperature and pressure have been optimised for maximum conversion of PET to TPA. The product obtained is characterised by recording FTIR and melting point.

Keywords: Chemical Recycling, High Pressure Autoclave, Hydrolysis, PET, TPA

INTRODUCTION

Nowadays, polyethylene Terephthalate (PET) is commonly used to manufacture water bottles having excellent water and moisture barrier properties. PET is commonly used in making mineral water bottles and soft drinks or carbonated beverage bottles. Huge use of Polyethylene Terephthalate generates considerable amount of waste. Consequently, gigantic amount of synthetic waste is generated; bulk of the waste is disposed in landfills or incinerated. The process of dumping synthetic waste in the land fill is not environmental friendly solution since polyethylene terephthalate (PET) bottles are not biodegradable. Since, the cost of the disposal of waste is increasing without a break, owing to the limited capacity of the landfills is causing soil pollution. As a result, it is of a great interest to chemically recycle and later reuse the materials. Primary recycling of Polyethylene Terephthalate (PET) converts the waste into other products of the virgin polymer. Number of researchers (Chen *et al.,* 1991, Vaidya *et al.,* 1989, Baliga *et al.,* 1989, Mandoki *et al.,* 1986, Paszun *et al.,* 1997 and Reimschuessel 1980) have dedicated their work to find alternative methods for the recycling of polyethylene Terephthalate (PET), polyurethane (PU) foam, Polyamide (PA). The recovery of the product of depolymerisation of polyethylene terephthalate can be used for conversion of PET (Mishra *et al.,* 2002 and Mishra *et al.,* 2003). We have studied the depolymerisation of PET by alkaline hydrolysis process adding pyridine as a catalyst using the high pressure autoclave.

MATERIALS AND METHODS
Materials
Sodium hydroxide, ethyl alcohol, hydrochloric acid and pyridine were used as such obtained by BDH chemicals. Waste mineral water bottles were used as a source of Polyethylene Terephthalate (PET). The bottles were cut into the size of 1 cm³ (one by one centimetre) into square shape. Amar Equipment Pvt. Ltd manufactured 0.5 L capacity High Pressure Autoclave was used to study.
Methods
Experimental: 10 g of Polyethylene Terephthalate (PET) waste, which was cut into the 1 cm³ square shaped pieces, 8 g of Sodium hydroxide and 250 ml of distilled water, were charged into the reaction

vessel of HPA. The reaction mixture was homogeneously mixed by string at the rate of 1000 RPM. The reaction was carried out at the temperatures 150, 180, 200, 220 °C the autogeniuos pressure was recorded at respective temperatures. The reaction was carried for the 120 minutes reaction time. After the completion of reaction time, the vessel was cooled suddenly by circulating cold water in the vessel through inner coil. The reactor vessel was opened by removing the collar when the vessel got completely cooled,

Chemical recycling of 10 g PET by alkaline hydrolysis was carried out by refluxing the PET along with water and catalyst for several time intervals. Amount of Sodium Hydroxide was varied for its optimization as 4, 6, 8 gram. The same reaction was carried out using with and without catalyst. 4 ml of pyridine as a catalyst and 8 gram optimized amount of sodium hydroxide were use in the reaction. The reaction was reflux for 120 minutes. The reaction mixture was cooled down on it's own. Once the reaction mixture got cooled, it was collected in the beaker. The TPA is precipitated as a white solid by adding concentrated hydrochloric acid in a reaction mixture. The product obtained was filtered, dried and weighed. The melting point of the product (TPA) was recorded as 249° C. TPA obtained is characterised by recording FTIR as shown in figure 1.
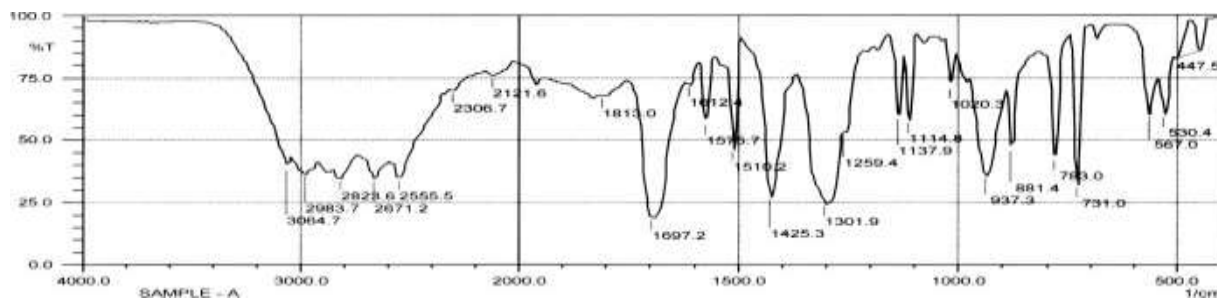

Figure 1: FTIR spectra of TPA

High Pressure Autoclave: Amar Equipment Privet Limited, Kurla, Mumbai was founded in 1974. Ever since 1974, Amar has built its foundation on a strong technical team with a blend of vast experience & technological and commercial understanding. Figure 2 shows assembly of high pressure autoclave.

Figure 2: High Pressure autoclave assembly

Applications: HPA is used for high-pressure high-temperature chemical reactions like hydrolysis, alkylation, amination, bromination, carboxylation, catalytic reduction, chlorination, dehydrogenation,

esterification, ethoxylation, halogenation, hydrogenation, methylation, nitration, oxidation, ozonization, polymerization, sulphonation etc. Prominently HPA is use in R&D centers of pharmaceuticals, dyes, chemical, fertilizers, paints, oils, agrochemical, and petrochemicals industries. It has been used by the colleges, research institutes, and defense organizations. HPA is used, where high pressure reactions and testing is carried out. Some specific use of HPA is listed below.

- To design new molecules, chemicals and to study the reaction parameters
- To manufacture the chemicals in small quantities in batch or continuous mode
- For synthesizing hydrogenation, acetylation, epoxidation, Grignard reaction, nitration, acylation, photochemical reactions, chemical recycling and quality control & process improvements
- For supercritical CO solvent extraction reaction, also for reaction calorimetry to study heat of reaction
- For high pressure storage and transfer of gas / liquid / slurries, acid digestion and Gas hydrate formation.

Salient Features: HPA is available as Stirred and non-stirred reactors, pressure vessels with the volume ranging from 5 ml to 500 ml for research laboratories and for industrial and pilot plant it is up to 2000 liter. The vessel is made up of SS-316/316L, Hastelloy B/C, Monel, Inconel, Nickel, Titanium, Tantalum lined, Zirconium etc. The maximum design pressure is up to 1000 psi and temperatures up to 650 °C. It is provided with high torque maintenance free zero leakage magnetic drive coupling. It is meant foe to design complete pilot plant with automatic temperature, pressure, RPM, motor torque/ current, liquid and gas. It is also provided with condenser for distillation or reflux, thermic fluid heating and cooling system etc. It is fully automated PC controlled high pressure systems and completely flame, explosion proof, ATEX certified systems.

RESULTS AND DESCUSSION

i. Reaction mechanism: The reaction mechanism for depolymerization of polyethylene terephthalate to terphthalic acid is represented as given below.
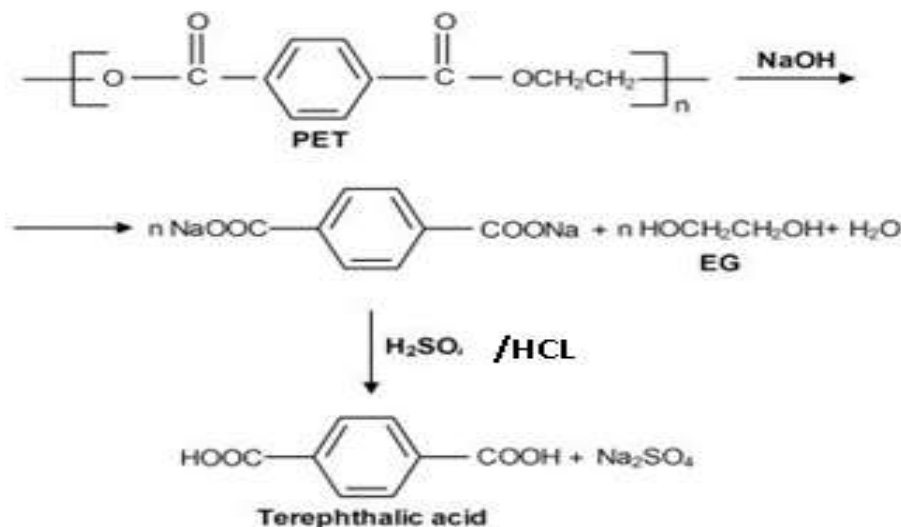
ii. Measurements of Autogeniuos pressure: The pressure exerted by the steam of water in the closed vessel is referred as autogeniuos pressure. The autogeniuos pressure was recorded at different temperature when three fourth of the vassal of 500ml capacity is filled by water. Table 1 and Figure 2 show variation of pressure with temperature. It is evident from the figure the trend is same as theoretical.

Table 1: Variation of Temperature versus Pressure

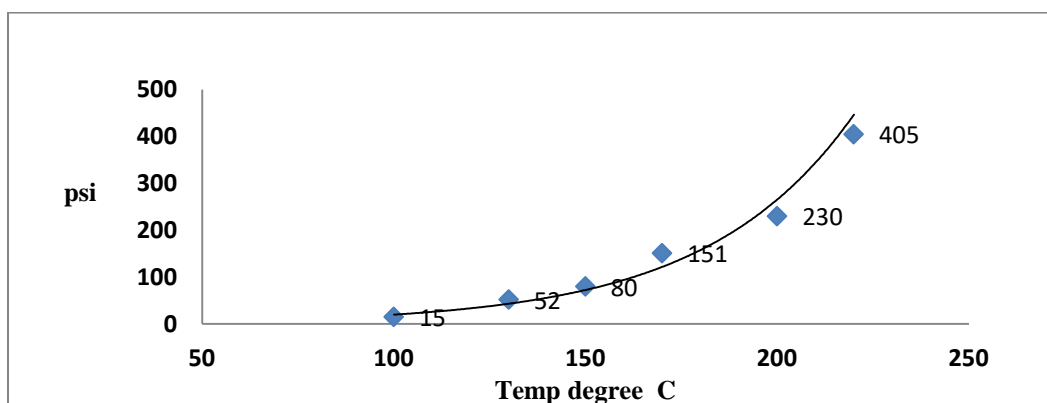| Temp ^{0}C	100	130	150	170	200	220
Pressure (psi)	15	52	80	151	230	405
Pressure (bar)	2	3.8	7.0	10.2	16.0	28.0

Figure 2: Variation of Temperature versus Pressure

iii. Conversion of PET to TPA using HPA: The variation of percenatge yield for the conversion of PET into TPA with temperature at respective autogenious pressure is as shown in figure 3. It is evident from the graph that, % yield is 69 at 150°C and at autogenious pressure 80 psi and then it suddenly increases up to 89% at 180°C. Thereafter there is no significant increase in % yield. It reaches to maximumm to 95% at 220°C and at auotgenious pressure 405 psi.
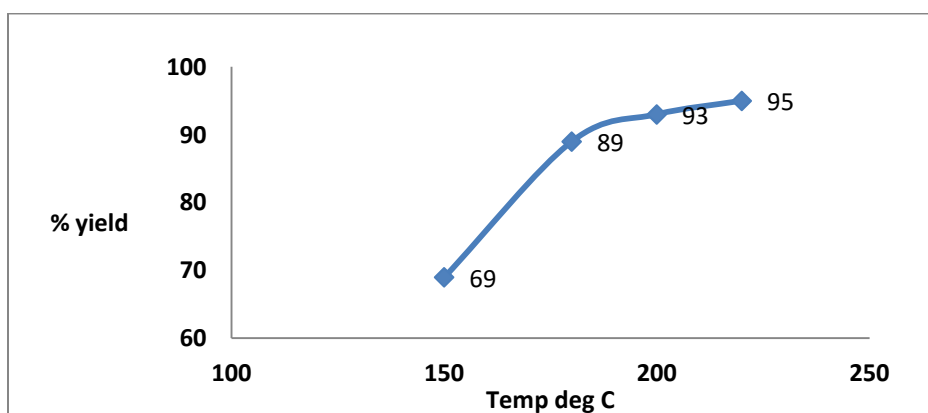

Figure 3: Variation of % yield versus Temperature

iv. Conversion of PET to TPA by using reflux method (with and without catalyst): Figure 4 indicates same trend of increase of percentage yield with and without catalyst. But at every temperature increase in percenatge yield is more when catalyst was used than without catalyst.

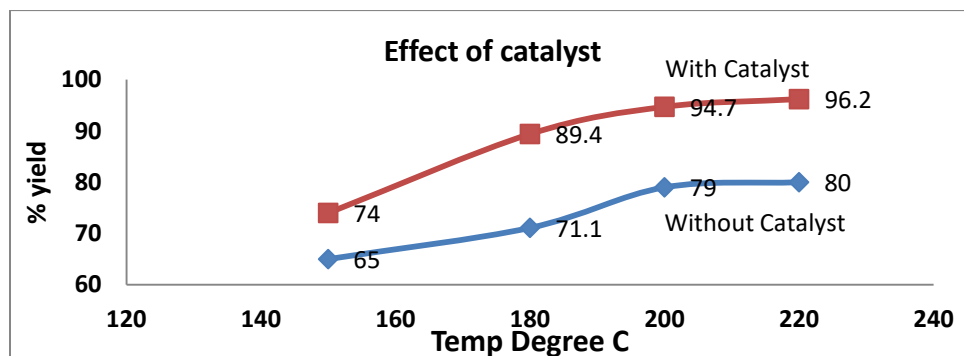The maximum percentage yield at 220°C was found to be 96.2 and 80 % with and without catalyst respectively.

Figure 4: Variation of % yield versus Temperature (with and without catalyst)

CONCLUSION
1. High pressure autoclave is use for study of depolymerisation of plastics waste
2. Calibration curve is set up by measuring autogenious pressure at different temperature.
3. Maximum percentage yield for obtaining monomer TPA by depolymerization reaction is 95 % at 220°C using high pressure autoclave.
4. Maximum percentage yield for obtaining monomer TPA by depolymerization reaction is 96.2 and 80 % with and without catalyst respectivly at 220°C using reflux method.
5. 8 gram of sodium hydroxide for depolymerization of 110 gram of PET has been optimized.

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Progress in Chemical Science Research

Vol. 3

Study on Catalytic Depolymerization of Polyurethane Using Zinc and Lead

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ABSTRACT

In the present work, Polyurethane (PU) waste was depolymerized by neutral hydrolytic process in presence of zinc acetate and lead acetate as catalysts. Extent of depolymerization of polyurathene was studied by measuring the amine value of the product. The ideal quantity of catalysts, including zinc acetate and lead acetate, was discovered to be 1g. For the depolymerization of PU foam reaction, zinc acetate was a more potent catalyst than lead acetate. Based on the amine value and residual weight of the depolymerized product, the velocity constant was obtained and found to be in order of 10^{-3} min^{-1}; and the reaction was found to be first order. The energy of activation and frequency factor determined by Arrhenius plot were 36.86 kJ $mole^{-1}$ and 1.349 x 10^2 min^{-1} respectively. The enthalpy of activation at 150^0C, 180^0C, 200^0C, and 240^0C was recorded as 43.89, 44.39, 44.72 and 45.37 $kJmole^{-1}$ respectively. The entropy of activation at the respective temperatures was recorded as 10.37 x 10^{-2}, 9.79 x 10^{-2}, 9.45 x 10^{-2} and 8.84 x 10^{-2} kJ $mole^{-1}$.

Keywords: Energy of activation; depolymerization; kinetics; free energy of activation.

1. INTRODUCTION

The increased applications of Polyurethane (PU) materials give rise to a higher amount of wastes, which have a devastating effect on the earth in turn; therefore, the management of end-of-life wastes is one of the most important issues in the modern world [1-3]. The depolymerization of PU into amines and polyols is accomplished through the well-known mechanisms of hydrolysis, glycolysis, and aminolysis. Primary polyols, carbon dioxide, and the amine of conjugate isocynate are all byproducts of the hydrolysis of polyurethane. Alcohol is used as the cracking agent during the alcoholysis depolymerization of PU foam. While the chemical reactions involved in alcoholysis and hydrolysis are slightly different, they both operate similarly. Different routes are to blame for receiving very different goods. PU waste output rises together with polyurethane manufacture.

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Traditional methods of destroying this waste are not particularly acceptable. Burning of polyurethane waste sends oxides of nitrogen, hydrocyanic acid, carbon dioxide and other toxic compounds in the atmosphere. When it is buried, it is broken down by action of water to give urea. This process causes pollution of air and water in environment. The most effective method of polyurethane waste processing is glycolysis, based on thermo-chemical interaction between polyurethane and hydroxyl containing compounds [4,5]. Literature survey indicates, glycolysis process can be used to resolve the disposal problems of PU waste and with obtaining high quality of polyols. Kouji et al. [6] have indicated the method to design a comfort path of PU recycling. The degradation of polyurethane foam was carried out with diethylene glycol as a solvent and sodium hydroxide as the catalyst [7]. In the present work PU waste was depolymerized by neutral hydrolytic process in presence of zinc acetate and lead acetate as catalysts. The amount of catalyst was optimized.

2. EXPERIMENTAL

5 g PU foam, 300 ml water and catalyst of definite amount were charged in the reaction vessel. The reaction was carried out for 60 minutes at temperature 180^0C by varying the amount of catalysts from 0.4-1.2 g. Further the reaction was carried out using optimized amount of catalyst by varying the temperature from 150^0, 180^0, 200^0 and 240^0 C for the reaction time of 30, 45, 60 and 90 minutes. After the specified reaction time the vessel was cooled suddenly by circulating cold water through the inner coil. The vessel was opened by removing collar of reactor of high-pressure autoclave. Obtained product was cooled to room temperature. The reactant was filtered using Whatman filter paper. Amine values were determined using one gram of depolymerized product by AOCS method.

$$\text{Amine value} = {}_{(}\text{Equivalence point x N x 56.1) / W}$$

W = Weight of sample in gram.
N = Exact Normality of HCl

The depolymerized product was taken for recovery of value added product. It was recovered by adding potassium hydroxide into depolymerized product. The recovered salt was characterized by recording FTIR spectra on Hyper FTIR of SHIMANDZU make.

3. RESULTS AND DISCUSSION

3.1 Optimization of Catalysts Concentration for Depolymerization of PU Foam

Depolymerization of PU foam waste was studied using lead acetate and zinc acetate as catalysts. Both the catalysts were taken from 0.4 g to 1.2 g using, 5 g of PU foam waste in 300 ml water at temperature of 180^0C and autogenious pressure of 160 psi for reaction time of 60 minutes. Results show an increase in percent conversion of PU foam waste with increase in amount of the catalysts,

100% and 71% PU foam was found to be depolymerized by taking 1g of zinc acetate and lead acetate respectively. Further increase in amount of catalyst results decrease in percent conversion in case of lead acetate (Fig. 1). Hence the optimized amount of catalyst for the depolymerization reaction is found to be 1 g for both zinc acetate and lead acetate. Decrease in percent conversion beyond optimized amount is might be due to occurring of reverse reaction at higher concentration of catalyst.
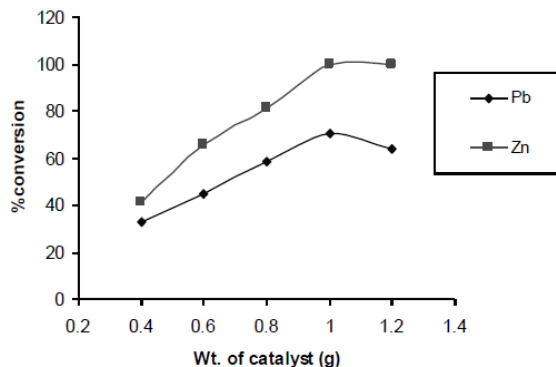

Fig. 1. Depolymerization of PU foam waste with catalysts at180^{0}C and 160 psi autogenious pressure for the reaction time 60 minutes

To measure the depolymerization value of PU foam waste, amine value of depolymerized product was determined (Fig. 2). Behavior of the Fig. 2 is exactly same as that of Fig. 1. It shows an increase in amine value with increase in concentration of catalyst up to 1g and thereafter there is slight decrease in amine value for both the catalysts. It is also evident from this figure that the optimum weight of catalyst for depolymerization of 5 g PU foam waste is 1 g.
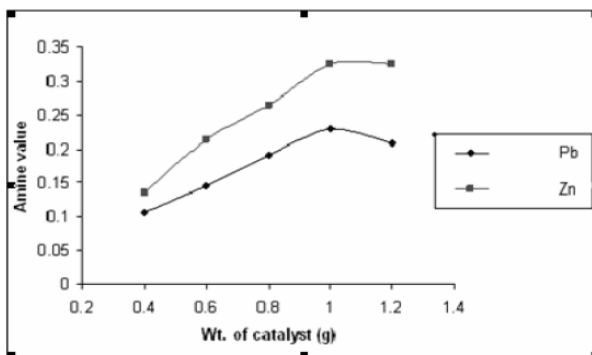

Fig. 2. Effect of catalyst on depolymerization of PU foam waste at 180^{0}C by measuring the amine value

Fig. 3 shows the results of amine values of depolymerized PU foam with increasing reaction time from 30 minute to 90 minute at 1 g of optimized amount of catalyst at 180^0C temperature and at autogenious pressure of 160 psi . The amine values increase with increase in time from 30-90 minute of reaction time in case of zinc acetate and uncatalysed reaction. The increments in amine value are recorded as 0.2246 for 30 minute reaction time and 0.3358 for 90 minute reaction time for zinc acetate. These values are highest for respective reaction times of lead acetate and uncatalysed reactions. It is worthy to mention that the lead acetate shows increase in amine value up to 0.3011 for 60 minute and thereafter it decreases to 0.2884 for 90 minutes of the reaction time. But it is also evident that zinc acetate catalyst is an effective catalyst for the depolymerization of PU foam waste, as the amine value is always greater at any instant as compared to that of lead acetate and without catalyst. Hence in this study we have undertaken the optimized amount (1 g) of zinc acetate catalyst for all the depolymerization reactions.
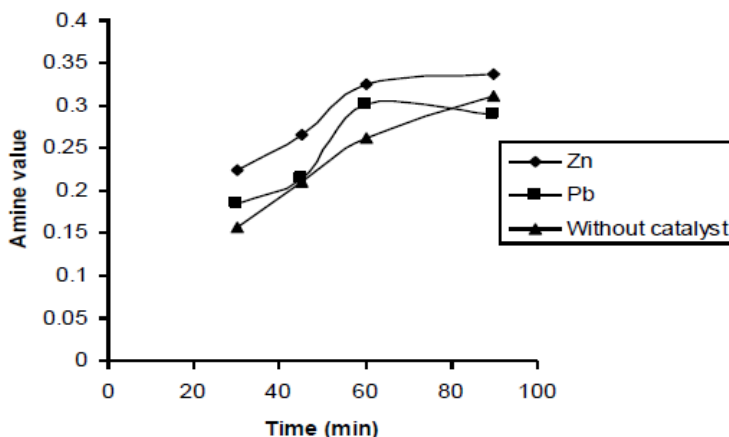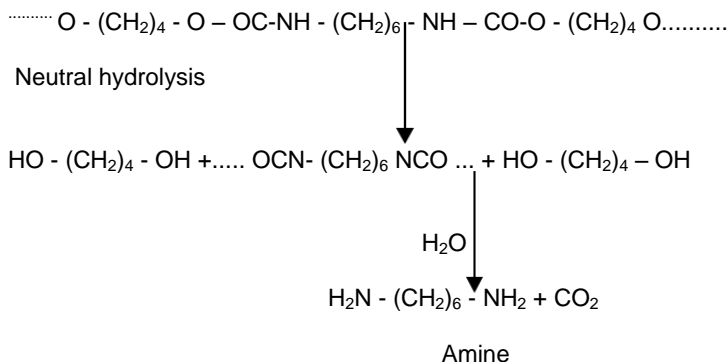

Fig. 3. Effect of reaction time on depolymerization of PU foam waste with and without catalysts at 180^0C reaction temperature

3.2 Kinetics of Depolymerization of PU Foam Waste

PU foam on depolymerization breaks into fragments of NCO groups. The rate of depolymerization depends on number of NCO groups produced.

The NCO-groups produced in depolymerization reaction react with water to form amine; hence, rate of depolymerization is decided by measuring amine value at various time intervals.

The mechanism of the reaction can be represented as,

.... O - $(CH_2)_4$ - O – OC-NH - $(CH_2)_6$- NH – CO-O - $(CH_2)_4$ O..........

Neutral hydrolysis

HO - $(CH_2)_4$ - OH +..... OCN- $(CH_2)_6$ NCO ... + HO - $(CH_2)_4$ – OH

H_2O

H_2N - $(CH_2)_6$ - NH_2 + CO_2

Amine

The rate of depolymerization was determined by measuring the amine value of the reaction product as well as residual weight of the PU foam waste. Amine value at zero time measures amine groups available at zero time. Similarly the amine value at $t = \infty$ is a measurement of total amine groups present after the completion of reaction. The first order kinetic model proposed by auothers [8] was found to be fit for our study using different catalyst,

$$k = 1/ t [- \ln (1-Amine\ value)] \tag{1}$$

Equation (1) was used to calculate the velocity constant of depolymerization of PU foam on the basis of measurement of amine value. The values obtained for velocity constant at temperature 180^0C and at autogenious pressures of 160 psi were plotted against the weight of the catalysts. Fig. 4 shows velocity constants, which are obtained maximum (6.56×10^{-3} and 4.36×10^{-3} for zinc acetate and lead acetate respectively) for 1 g of the catalysts. The values of velocity constants are recorded as 6.56×10^{-3} and 3.88×10^{-3} for zinc acetate and lead acetate at 1.2 g of catalysts. Thus it can be concluded that below optimum amount of the catalysts the reaction is slow in case of both the catalysts, and above the optimum amount the reverse reaction is there in case of lead acetate.
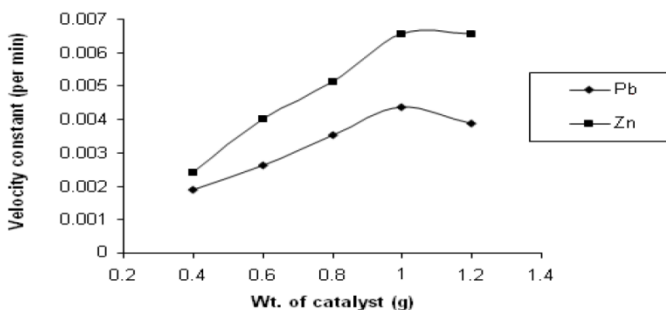

Fig. 4. Effect of amount of catalyst on velocity constants (min^{-1}) for depolymerization of PU foam waste at 180^0C

Velocity constants of the depolymerization reactions are also determined by using residual weight left after the complete reaction by equation as given below,

$$k = \frac{2.303}{t} \, log \frac{c_0}{c_t} \tag{2}$$

Where $[C]_O$ is the concentration of the reactant at time t = 0 and $[C]_t$ is the concentration of the reactant at time t. It is observed that the velocity constants obtained by equation (1) and (2) are identical and are of the order of 10^{-3} min.$^{-1}$

Fig. 5 shows an increment in the velocity constant with temperature at all the time intervals. An increase in velocity constants of the reactions is due to higher kinetic energy of the molecule obtained by thermal agitation. However, as reaction time increases the velocity constant decreases. The velocity constants increase for the 30 minutes reaction time but subsequently, at 45, 60 and 90 minutes, these values decrease successively for all respective temperatures, since the product of the reaction was not removed. It might be due to a Le-Chatelier effect [9].
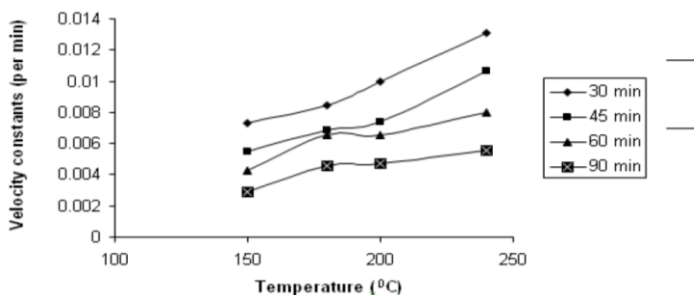

Fig. 5. Effect of temperatures on velocity constants at different reaction time interval using optimized zinc acetate catalyst

The amine value at 150^0 C remains almost constant (0.22) at various time intervals Fig. 6. While variation in amine value increases up to 0.32 with time at 180^0 C and 200^0C. Further the amine values remain almost constant at these temperatures. The degree of depolymerization is significant at 240^0C. Initially up till 45 minutes reaction time, amine value gradually increases to 0.38 and thereafter it remains constant.

As per the reaction mechanism shown earlier, each chain scission utilizes one water molecule to form one each of amine and glycol end groups. Therefore, the progress of the reaction was studied by measuring concentrations of amine groups after the definite reaction time.
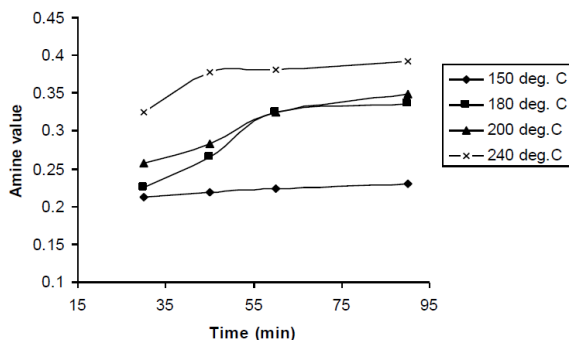
Fig. 6. Effect of reaction time for depolymerization on amine value at different temperatures using optimized zinc acetate catalyst

The rate of amine end group formation may be expressed as,

$$d[-NH_2] / dt = k \,[PU][water] – k' \,[-NH_2]\,[OH] \qquad (3)$$

Therefore, the equilibrium constant Ke is given as,

$$Ke = k / k' \qquad (4)$$

Where,

k   = the rate constant of forward reaction (depolymerization),
k'   = the rate of reverse reaction (condensation),
[PU]   = concentration of polyurethane foam,
[Water] = concentration of water,
$[-NH_2]$ = concentration of amine end group,
[OH]   = concentration of hydroxyl end group

Equations 4 was used to calculate rate constant of reverse reaction and hence the equilibrium constants were determined.The results of comparative study of velocity constants with and without catalyst of depolymerization reaction at temperatures 100^0,150^0, 180^0,200^0,and 240^0 C and at autogenious pressure were studied by Mishra et al[6]. The values of velocity constants obtained by measuring residual weight and amine values were found to be identical and these are in order of 10^{-3} min^{-1}. It indicates that the mathematical first order kinetics model proposed by Mishra et al[6] is justified.

3.3 Thermodynamics of Depolymerization of PU Foam Waste

Arrhenius plot was used to elucidate the activation energy. The energy of activation for depolymerization of PU foam waste was obtained by studying the depolymerization reaction at the temperatures ranging from 150^0 C to 240^0 C. The Arrhenius plot was drawn using the values of ln k versus 1/T. The slope of

Arrhenius plot was found to be 4433.6 unit which was used to calculate the energy of activation. The energy of activation for depolymerization of PU foam waste was calculated as 36.86 kJ mole^{-1} (Fig/ 7). The intercept of Arrhenius plot is 4.9046 units, which is used to determine the frequency factor of the reaction, and it is found to be 1.349×10^2 min^{-1}. It shows that reactant molecules collide with each other with the frequency 1.349×10^2 min^{-1}.
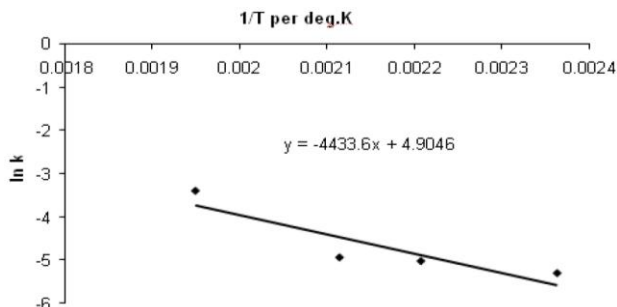

Fig. 7. Arrhenius plot of variation of velocity constant (ln k) versus reciprocal of absolute temperature (1/T)

4. CONCLUSION

The enthalpy of activation at various temperatures was obtained using the equation [10] $\Delta H = E_a + 2RT$. The minimum value of ΔH (43.89 kJ mole^{-1}) was recorded for 150^{0}C reaction temperature, while maximum (45.37 kJ mole^{-1}) for 240^0 C; on recording the entropy, the values are vice versa. It is evident from the values that as temperature increases, enthalpy of activation increases while entropy of activation decreases. The decrease in entropy with increase in temperature is due to higher rate of depolymerization so that conversion of PU foam waste obtained at early time.

Kinetic and thermodynamic parameters such as velocity constant for forward reaction (k), velocity constant for reverse reaction (k'), thermodynamic equilibrium constant (Ke) and Gibbs free energy (ΔG) at temperature 180^{0}C and for 60 minutes reaction time are summarized in Table 1. Results show that as the amount of the catalyst increases the Gibbs free energy of activation also increases up to 1 g of catalyst. Hence this amount is an optimum amount for depolymerization of PU foam waste. Same behavior is observed for zinc acetate catalyst, however the values of k, k'and ΔG can not be evaluated since residual weight was not available due to complete disappearance of the reactant. However in case of lead acetate, the value of Ke is recorded in decreasing order from 32.38 to 5.99 for 0.4 to 1.0 g .Thereafter the value of Ke increases up to 7.58 for 1.2 g of lead acetate catalyst. Likewise value of ΔG increases up to 1 g of catalyst and further decreases for 1.2 g of lead acetate. Thus it is concluded that reversible reaction becomes more pronounced beyond 1 g of lead acetate as catalyst.

Table 1. Kinetic and Thermodynamic parameters for depolymerization of PU foam waste at 180^0 C using different catalysts

Weight(g)	Lead acetate catalyst				Zinc acetate catalyst			
	$k \times 10^{-3}$ min^{-1}	$k' \times 10^{-4}$ min^{-1}	Ke	$-\Delta G$ $kJ\ mole^{-1}$	$k \times 10^{-3}$ min^{-1}	$k' \times 10^{-4}$ min^{-1}	Ke	$-\Delta G$ $kJ\ mole^{-1}$
0.4	1.878	0.57	32.38	13.097	2.71	1.393	19.45	11.177
0.6	2.627	1.572	16.71	10.605	4.31	6.019	7.16	7.413
0.8	3.527	3.799	9.28	8.390	5.316	11.210	4.38	5.562
1.0	4.356	7.262	5.998	6.746	-	-	-	-
1.2	3.878	5.110	7.586	7.631	-	-	-	-

COMPETING INTERESTS

Author has declared that no competing interests exist.

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Biography of author(s)

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He is a Professor in Chemistry and Principal of KCE Society's Post Graduate College of Science, Technology and Research, Jalgaon. He has 37 year teaching experience at PG and UG level and 30 years in research. He has completed his doctorate degree from KBC North Maharashtra University, Jalgaon in the subject of Chemical Recycling of Plastic Waste in the year 2004. He has to his credit 33 publications in National and International research journal of repute. 3 students have awarded Ph.D. and 02 were awarded M. Phil. under his guidance. He was the chairman of steering committee for NAAC re-accreditation process of the college and the College is awarded as "A" grade with highest CGPA score in subsequent two cycles. He is the author of 35 text books in physical and analytical chemistry useful especially for the students of under graduate. He has worked as visiting faculty to several colleges and also at Department of Chemistry of KBC North Maharashtra University, Jalgaon to deliver the lectures on the topics such as Quantum Chemistry, Statistical Thermodynamics, Molecular Spectroscopy, Chemical Kinetics etc. He visited Thailand, Malaysia, Estonia, Denmark, Finland, Norway, Estonia and Sweden to attend and present research papers in conferences and study the educational systems of abroad.

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