

# **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 autogenious 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**

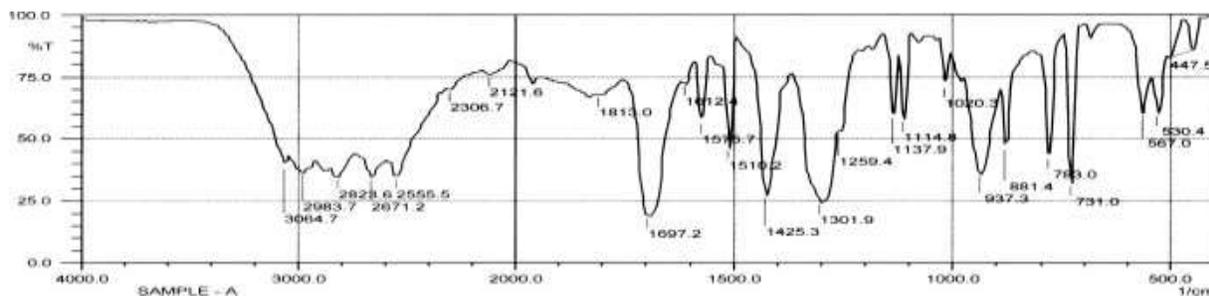
Sodium hydroxide, ethyl alcohol, hydrochloric acid and pyridine were used as such obtained by BDH chemicals. Waste mineral water bottles were used as a source of Polyethylene Terephthalate (PET). The bottles were cut into the size of 1 cm<sup>3</sup> (one by one centimetre) into square shape. Amar Equipment 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 cm<sup>3</sup> 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 autogenous 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 used in the reaction. The reaction was reflux for 120 minutes. The reaction mixture was cooled down on its 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 Pvt Ltd, 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,

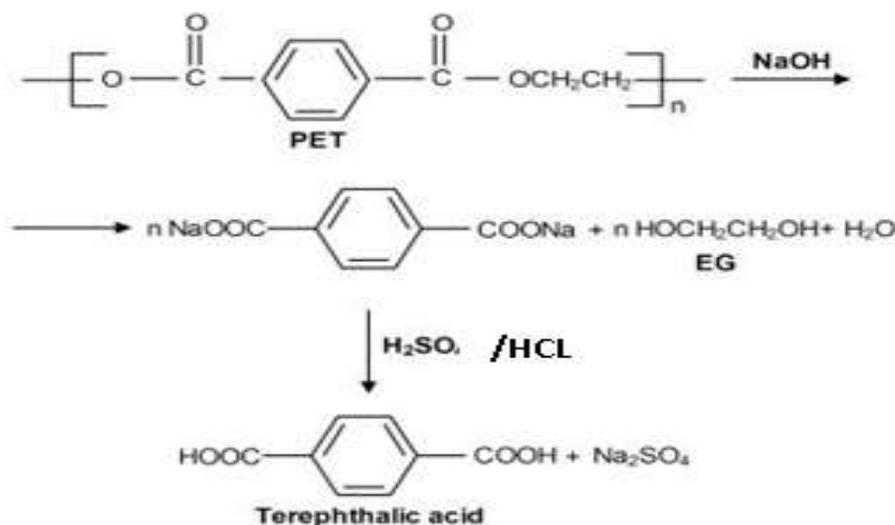
esterification, ethoxylation, halogenation, hydrogenation, methylation, nitration, oxidation, ozonization, polymerization, sulphonation etc. Prominently HPA is use in R&D centers of pharmaceuticals, dyes, chemical, fertilizers, paints, oils, agrochemical, and petrochemicals industries. It has been used 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

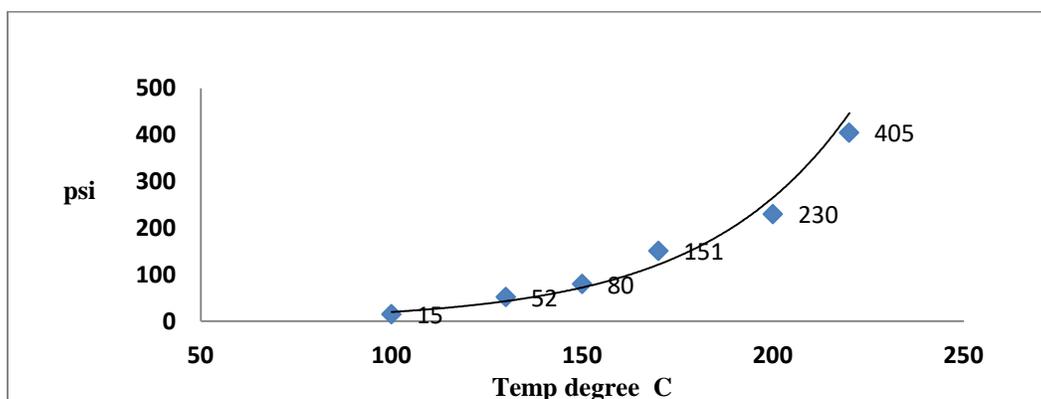
**i. Reaction mechanism:** The reaction mechanism for depolymerization of polyethylene terephthalate to terphthalic acid is represented as given below.



**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.

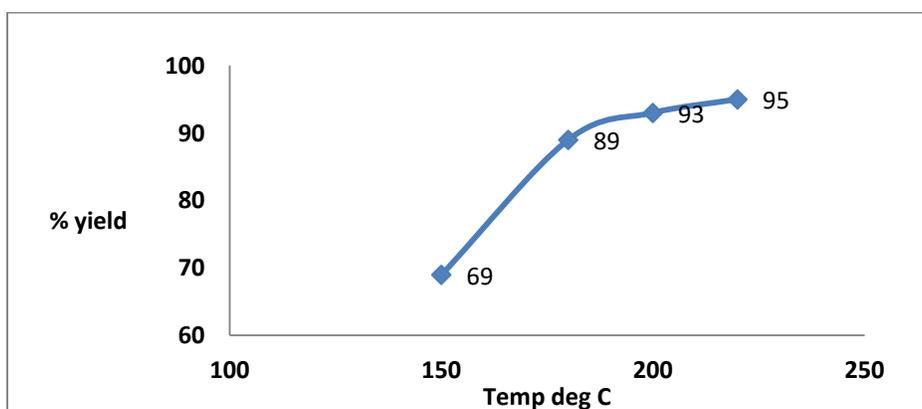
**Table 1: Variation of Temperature versus Pressure**

<b>Temp °C</b>	100	130	150	170	200	220
<b>Pressure (psi)</b>	15	52	80	151	230	405
<b>Pressure (bar)</b>	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 percentatge yield for the conversion of PET into TPA with temperature at respective autogenous pressure is as shown in figure 3. It is evident from the graph that, % yield is 69 at 150°C and at autogenous 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 autogenous 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 percentatge 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.

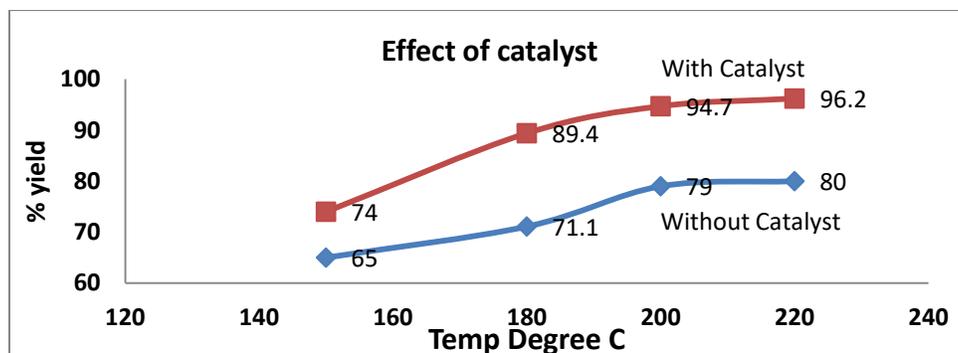


Figure 4: Variation of % yield versus Temperature (with and without catalyst)

## CONCLUSION

1. High pressure autoclave is use for study of depolymerisation of plastics waste
2. Calibration curve is set up by measuring autogenous 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 respectively 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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