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STUDY OF THE PROCESS OF AMINOLYSIS OF SECONDARY POLYETHYLENETEREPHALATE WITH MONOETHANOLAMINE WITHOUT THE PARTICIPATION OF A CATALYST AND ANALYSIS OF THE OBTAINED PRODUCT

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Abstract: In this study, secondary polyethylene terephthalate (PET), an industrial waste, and monoethanolamine were treated in a ratio of 1:2.5 without the presence of a catalyst. The aminolysis process was carried out stepwise at 100-190 0 C for 5 hours. The resulting product fractions were separated and crystallized with twice their mass of water. The resulting product was separated into fractions using a vacuum filter. The filtrate was found to have a high yield of bis(2-hydroxyethyl)terephthalamide (BHETA) of 80%. The product was analyzed using IR spectroscopy. This article proposes an energy-efficient, environmentally friendly and catalyst-free method for obtaining value-added products from PET waste by recycling.

Keywords: secondary polyethylene terephthalate (PET), monoethanolamine, BHETA, catalyst.

Introduction. Polyethylene terephthalate (PET) is a versatile engineering plastic with excellent thermal and mechanical properties. It is a semi-crystalline thermoplastic polyester with high strength, transparency and safety. Its main applications include the textile industry and the production of video and audio tapes, X-rays, food packaging and especially water and soft drink bottles. Since the shelf life of PET waste (mainly from mineral water bottles and soft drink bottles) is very short, a large amount of waste stream reaches the final recipients every year and poses a serious environmental problem.

Literature review. Disposal of waste to landfills is becoming increasingly undesirable due to regulatory pressures, increasing costs, and poor biodegradability of common polymers. Therefore, PET waste should be recycled. Recycling is divided into three types: mechanical recycling, chemical recycling, and energy recovery. Mechanical recycling is the separation of the polymer from its associated contaminants and its easy processing into pellets using conventional melt extrusion. Chemical recycling is defined as a process that leads to the depolymerization of PET into monomers or other secondary

value-added products. Energy recovery usually refers to the recovery of the energy content of the plastic by incineration. Among the recycling methods, incineration faces strong societal resistance, and mechanical recycling can only be carried out on single-polymer waste streams. Therefore, chemical recycling of polymers is considered the most feasible method. [1,2] Chemical recycling is divided into the following types: hydrolysis, glycolysis, alkolysis, ammanolysis, aminolysis, and others [3-8]. The least studied of these is aminolysis. PET aminolysis was performed using a modern method, namely microwave irradiation. They performed PET:MEA in a ratio of (1:6) without the presence of a catalyst at high temperature (260 ^{0}C) with a radiation power of 100 W for 5 minutes [9]. PET aminolysis and ammanolysis were performed at room temperature for 3 to 45 days with and without the presence of a catalyst. Ammonium salts were used as catalysts. The properties of the resulting aminolysis and ammanolysis products were studied using a scanning electron microscope (SEM) [10]. Polyethylene terephthalate aminolysis was performed in different ratios of 1:1, 1:2.5, 1:4, 1:5.5, and 1:7 and in the presence of different catalysts sodium acetate and zinc acetate. The catalyst was added in an amount of 1.5% relative to PET. And it was found that the yield of BHETA formation was achieved in a high yield (81%) in the aminolysis synthesis lasting 3 hours at 160 ^{0}C in the presence of a catalyst zinc acetate in a ratio of 1:4. By adding hepatonic acid to the resulting BHETA, a plasticizer was obtained and synthesized with a conventional plasticizer DOP (dioctyl phthalate). The plasticizer synthesized had a high polarity due to the presence of amide and ether bonds[11]. The conditions for aminolysis of PET with hydrazine hydrate in conventional and modern (microwave) methods were studied. In the conventional method, PET and hydrazine hydrate were carried out in a ratio of 1:6 in the presence of a catalyst for 4 hours. In the microwave method, the aminolysis process was carried out in 10 minutes with the same substance ratios and in the presence of a catalyst. As a result, time and energy savings were achieved[12]. PET aminolysis was carried out in a 1:5 ratio without catalyst at 160 C for 2 h. The resulting product was analyzed using various FTIR, DSC, TGA and CHN methods. According to them, DSC and TGA analysis are reliable methods for the aminolysis process. However, the FTIR method shows that due to the presence of several purification steps to determine the conversion of the aminolysis reaction, overlapping of nearby functional groups was observed. Due to the presence of nitrogen in PET oligomers, the CHN method is unpredictable[13].

Experiments. Industrial PET waste was first separated from non-PET parts and cut into pieces of approximately 1-1.5 cm in size. In order to effectively separate the PET pieces from other impurities, they were placed in a hot water bath maintained at a temperature of 70–100°C. The PET pieces thus separated were thoroughly washed with distilled water and dried at a temperature of 60°C. Monoethanolamine was used for aminolysis. The aminolysis process was carried out in a three-necked flask equipped with a reflux condenser, thermometer, and stirrer under reflux conditions. PET:MEA was added at a molar ratio of 1:2.5 at different times without the presence of a catalyst, initially for 5 hours. The process was initially held at 100-115 °C for 1 hour. Then, it was held at 160-165 °C for another 1 hour, since the aminolysis process must be completely

homogeneous. After the synthesis was completely homogeneous, the temperature was raised to 190 °C and held at this temperature for 5 hours. The resulting product was crystallized at 90 °C for 30 minutes. The resulting product was filtered using a vacuum filter, fraction 1 was separated and placed in an oven at 80 °C for drying. The aminolysis product that passed through the filtrate was separated at room temperature for 24 hours and then vacuum filtered. The product remaining in the filter was placed in a desiccator to dry. We left the filtrate in the refrigerator for 24 hours. To calculate the material balance of our resulting products, we separated the unreacted MEA and the formed ethylene glycol by simple distillation of the remaining liquid after filtration (Table 1). The yield of BHETA formation was higher than 80% compared to PET.

Results. We performed the purification of the aminolysis product without the use of a catalyst and at a lower ethanolamine ratio than our previous work. As a result, we achieved a recovery of bis(2-hydroxy ethylene)terephthalamide (BHETA) of more than 86%. And the IR spectroscopic analysis of all fractions of the obtained BHETA product was recorded on the spectrometer "IRTracer-100" (SHIMADZU CORP., Japan, 2017) (Figures 1-3).

Below are the IR spectroscopic analysis results of the first, second and third fractions. It is seen that the IR spectra of all fractions have peaks at 3289-3368 cm $^{-1}$ -NH (amide), 1630 cm $^{-1}$ -C=O peak connected to the amide. 1560 cm $^{-1}$ is a characteristic peak for the secondary amide. 1443 cm $^{-1}$ -CH is a characteristic peak for the methylene group. 1376 cm -1 $^{-CH3}$, 1260 cm $^{-1}$ and 1302 cm $^{-1}$ -CN (amide) peaks can be seen. The peak at 1069 cm $^{-1}$ indicates the presence of this primary alcohol, while 868 cm -1 ^{is} characteristic for the para-substituted aromatic ring.

These peaks are specific to the BHETA molecule and indicate that the aminolysis process is complete.

Conclusion. Secondary polyethylene terephthalate was reacted with ethanolamine in a molar ratio of PET:MEA 1:2.5 for 5 hours without the presence of a catalyst. The products obtained were separated into 3 fractions. When the separated products were analyzed by IR spectroscopy, it was determined that they were all single substances. The functional groups formed were identified. In our future work, we will focus on increasing the product yield and studying it using NMR and DSC analysis methods.

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Table 1. Material balance for 5 hours (100 g) of substance in a ratio of PET:MEA 1:2.5:

Mass PET (per 100gr)	BETHA (gr)	MEA residue	EG residue	Dimer residue
55.7 grams	48.18 grams	1.52 grams	10.78 grams	33.15 grams

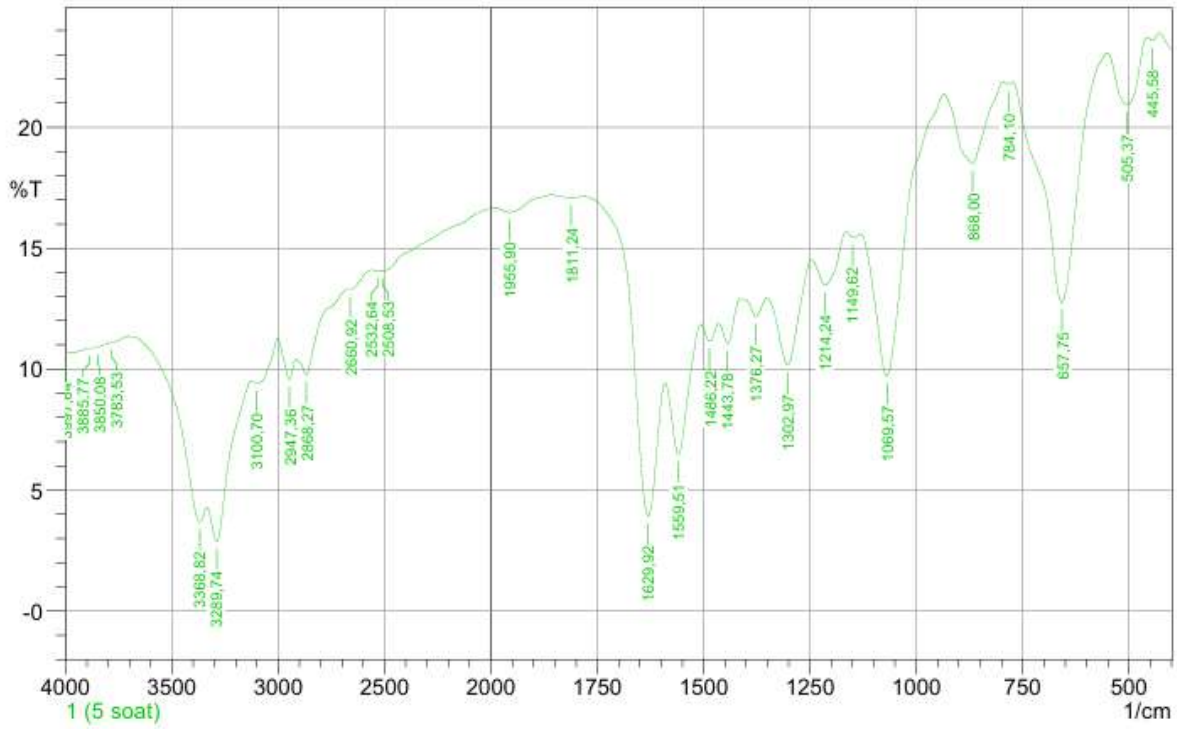

Figure 1. IR spectra of fraction 1 obtained by aminolysis

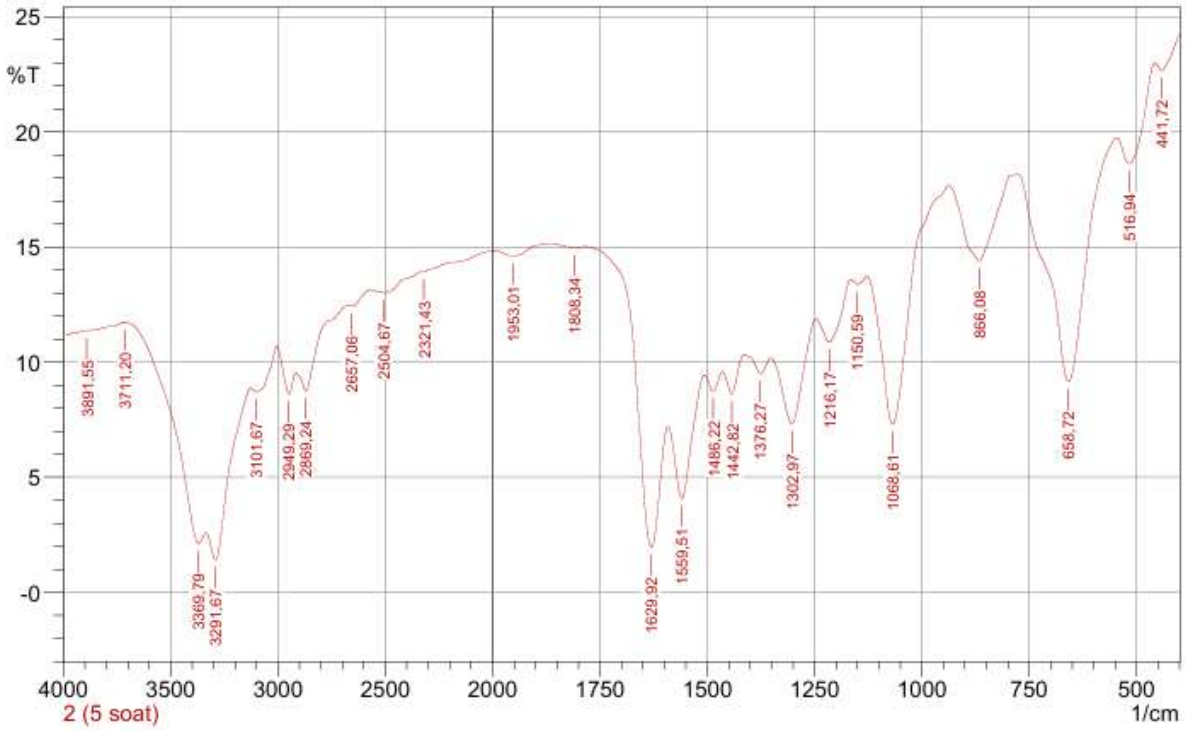

Figure 2. IR spectra of fraction 2 obtained by aminolysis

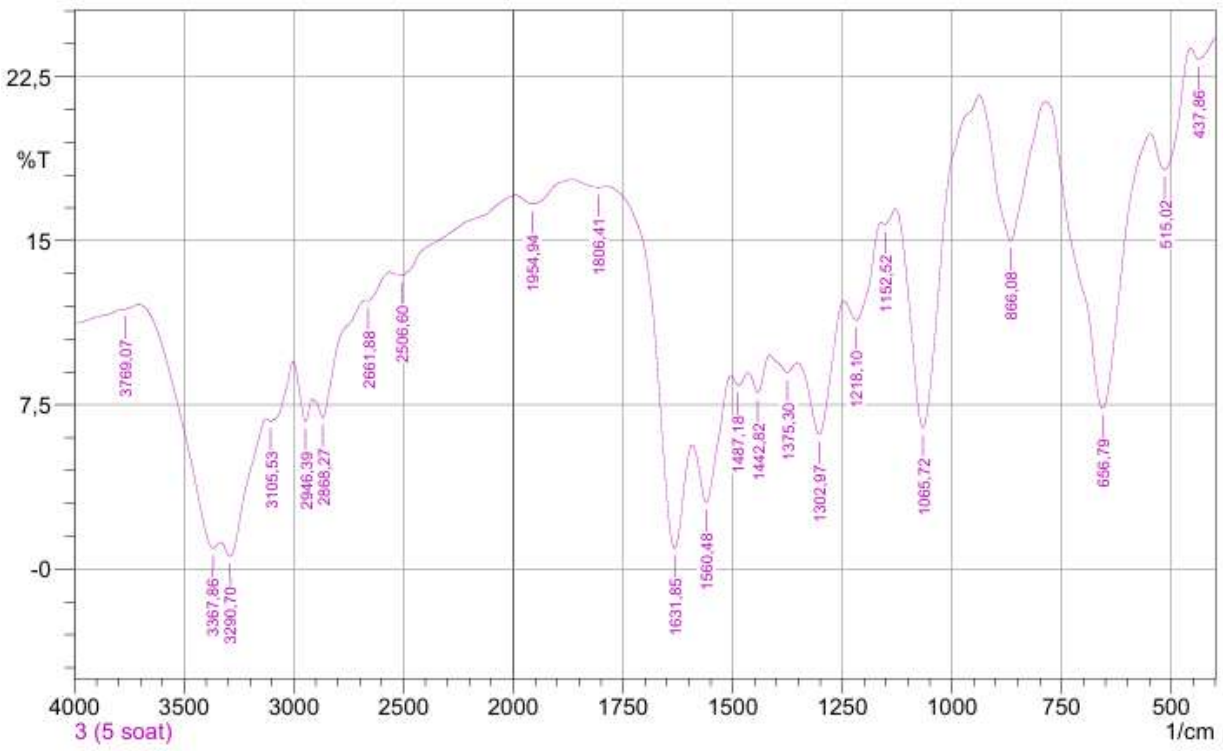

Figure 3. IR spectra of fraction 3 obtained by aminolysis

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