

## DETECTION OF MICROPLASTIC WASTE OF POLYETHYLENE TEREPHTHALATE (PET) IN SEAWATER AND CONSUMPTION SALT BY GLYCOLYSIS

<sup>1,2</sup>INDANG DEWATA, TRISNA KUMALA SARI<sup>1</sup>, DESY KURNIAWATI<sup>1</sup>, <sup>1</sup>KASMA WARNI, RAHMI AULIA MEILINDRI<sup>1</sup>, REZA ELVINDA<sup>1</sup> AND ATIKAH MUTHMAINNAH<sup>1</sup>

<sup>1</sup>Department of Chemistry, Universitas Negeri Padang, Indonesia

<sup>2</sup>Program Study of Environmental Sciences, Universitas Negeri Padang, Indonesia

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### ABSTRACT

Polyethylene Terephthalate (PET) is a type of plastic that is widely used for various needs such as beverage packaging bottles. However, PET waste can break down into microplastics and can be harmful if ingested by living things. This study purposed to detect the microplastic content of PET in seawater and consumption salt by glycolysis method. This method involves the process of depolymerization of PET to be a monomer of bis (2-hydroxyethyl terephthalate) (BHET). Determination of the optimum conditions was observed from the effect of ethylene glycol solvent, catalyst mass, and the reflux temperature of the glycolysis process. The optimum condition obtained is 30 ml of ethylene glycol (EG) solvent, 0.06 gram of Na<sub>2</sub>CO<sub>3</sub> catalyst mass for 1 h at a temperature of 196°C which obtained a yield of 72.63% BHET. The results showed the absence of PET in both seawater and saltwater samples.

**KEY WORDS:** PET, Glycolysis, Bis (2-hydroxyethyl terephthalate) (BHET), Microplastic.

### INTRODUCTION

The development of human needs in everyday life is a factor in a large number of plastics equipment. The use of plastic causes environmental pollution which is very dangerous for living things because plastic is very difficult to recycle and it is difficult to degrade naturally. Plastic materials that exist in the marine environment are the result of industrial disposal through inland waterways, wastewater streams, or carried by wind and tides. Pollution can come from the water close to residential areas. Human activities near the water cause pollution in the water (Dewata and Adri, 2018).

Most of the plastic waste dumped into the environment ends up in the ocean. The percentage of plastic waste that pollutes the sea reaches 60-80% of the total waste at sea. Plastic particles take a long time to degrade and hold for a very long time in marine environments. Meanwhile, the amount of this waste is increasing so that it piles up in the

water. Plastic waste that has been in the water for a long time will eventually be degraded into small particles called microplastics (plastic particles <5 mm in size). Their very small size allows microplastics to be accidentally digested by marine organisms. Organisms that accumulate in large numbers of microplastics will cause blockage in the digestive process, disrupt digestive processes and cause death (Foekema *et al.*, 2013).

PET is a type of plastic waste that is commonly found in the environment, especially in the ocean. The most common source of PET is single-use soft drink bottles. This plastic waste not only pollutes the oceans but can also break down into microplastics, which endangers the survival of aquatic life (Salamah and Maryudi, 2019).

The sea in Padang City has the potential to be contaminated by microplastics based on research (Saputra, 2018) saying that the dominant waste on the coast of Padang city is plastic waste at 36.85%. The amount of plastic waste at sea will certainly be

dangerous for the survival of marine life in Padang City. Plastic waste that is carried away by the current will break down into micro-sized particles are dangerous if swallowed by marine life, so we need to know about the existence of microplastics to educate the public about marine microplastic pollutants and minimize marine plastic waste disposal. Based on this, it is necessary to determine the content of microplastic waste types of PET in seawater in Padang city. Another possibility is salt contamination by PET waste. Seawater is pumped for evaporation to produce salt crystals on its surface. In the process of collecting salt crystals, salt which has physical differences will be separated into different containers according to the application and its use. The study shows that consumption of salt from the sea can be contaminated by microplastics because it is directly produced from seawater, so it is very important to know the presence of contamination such as in salt from seawater (Iñiguez *et al.*, 2017).

One way to overcome the problem of PET microplastic waste is to depolymerize PET into parts that have a simpler chemical structure (oligomers, dimers, and even back into their monomers) which no longer pollute nature and can be reused. Research on the chemical depolymerization of PET has been carried out with various solvolysis methods according to the solvent used, i.e. alcoholysis using alcohol, hydrolysis using water, and glycolysis using glycol. Alcoholysis using methanol (methanolysis) in the form of gas or liquid will produce dimethyl terephthalate (DMT) which has a disadvantage, i.e. the high volatility of methanol so that it can make technical difficulties in the process (Wang *et al.*, 2009). Hydrolysis of PET under acidic or alkaline conditions is carried out by (Baliga, 1989) producing terephthalic acid (TPA) which in the process can cause corrosion and pollution. The glycolysis method is a method that is often used because it is considered the most advantageous among the other methods. The advantage of the glycolysis method is the simple process, it can be carried out conventionally and the resulting bis (2-hydroxyethyl) terephthalate (BHET) monomer, and it can be reused for PET production thereby saving PET production costs.

According to the previous article (Rahmayanti, 2015), the rate of glycolysis without a catalyst runs very slowly so it requires a catalyst for the reaction to run fast. Of the several catalysts commonly used, the effective catalyst was zinc acetate with the

product of depolymerization yielding 78% yield (Sánchez and Collinson, 2011), although this catalyst is very effective for glycolysis, zinc acetate harms the environment because it is toxic. Therefore, the catalyst used is sodium carbonate, besides having almost the same effectiveness as zinc acetate, this catalyst is also more environmentally friendly. Therefore, in this study, detection of PET waste in seawater and salt samples using the glycolysis was carried out by determining the optimum conditions of the catalyst mass, amount of solvent, and temperature of reflux.

## EXPERIMENTAL SECTION

The equipment used in this study was a beaker, a two-neck flask, vaporizer cup, spatula, a set of reflux equipment, a funnel, a digital balance, a magnetic stirrer, an oven, a heater, burette, volume pipette, thermometer, and PerkinElmer's Fourier Transform Infrared Spectroscopy (FTIR) instrument. The materials used in this study are PET waste obtained from mineral drink bottles, seawater, filter paper, aluminium foil, HCl, ethylene glycol, distilled water,  $\text{Na}_2\text{CO}_3$ .

## METHODS

### Sample preparation of Polyethylene Terephthalate (PET)

Preparation of PET samples from PET plastic bottles is done by washing plastic bottles with detergent to clean and to remove impurities that stick to the surface of the bottles and rinse with distilled water. Washed bottles are then cut into small pieces. Then the sample was rinsed again with distilled water and dried in an oven at 60 °C for 1 hour (Setyowati and Atmaja, 2012).

### Determination of the optimum condition of ethylene glycol solvent in the glycolysis method

The sample of the prepared plastic bottle was weighed as much as 10 g and put into a three-neck flask equipped with a thermometer, condenser and magnetic stirrer, then was added ethylene glycol (EG) to the three-neck flask with a variation of the ratio of plastic bottles: EG (g: ml), *i.e.* 10:20, 10:30, 10:40, and 10:50 with the help of a sodium carbonate catalyst as much as 0.06 gram. The glycolysis reaction was carried out at a temperature of 196°C for 1 hour (López-fonseca *et al.*, 2011). Then, the flask

is transferred to an ice bath. The resulting product was added with distilled water in a hot state of 70 ml while stirring and then immediately the suspension was filtered using a vacuum filter. The product will separate into a solid phase and a liquid phase. The filtrate formed is a mixture of ethylene glycol, BHET, and a few dissolved oligomers. Furthermore, it is heated until a pellucid homogeneous mixture is obtained, then it is filtered again. The resulting filtrate was cooled at 5 °C for 16 hours to obtain BHET crystals. The result was filtered again and the BHET solids obtained were dried using an oven at 60°C until dry (Setyowati and Atmaja, 2012).

#### **Determination of optimum condition of catalyst mass of sodium carbonate**

The glycolysis reaction was carried out for catalyst mass variations of sodium carbonate of 0.02 g, 0.04 g, 0.06 g, 0.08 g, and 0.1 g which is refluxed for 1 hour at a temperature of 196 °C with the optimum condition of the ratio of plastic bottles and EG solvents in the previous experiment. The next procedure is the same.

#### **Determination of optimum condition of reflux temperatures**

The glycolysis reaction was carried out at various reflux temperatures, i.e 166 °C, 176 °C, 186 °C, and 196 °C with the ratio of plastic bottles and ethylene glycol solvent which produced the maximum amount of solids in the previous experiment.

#### **The Preparation and PET glycolysis of the seawater sample**

Seawater samples were filtered with a 5 mm filter to remove impurity and organisms. Then it is filtered again using filter paper. The residue on the filter paper is rinsed with seawater into a 20 ml beaker. Organic material will dissolve in acid at 80-90°C for 3 hours using concentrated HCl, then filter it with filter paper. The residue contained on the filter paper was covered with aluminium foil and dried at a temperature of 60°C (Desforges *et al*, 2012). Seawater samples are treated in the same way as PET glycolysis. based on the optimum conditions obtained.

#### **The preparation and PET glycolysis of the salt consumption sample**

The salt sample was weighed 10 gram into the evaporator plate then dried in an oven at 60 ° C for

1 hour. Seawater samples are treated in the same way as PET glycolysis. based on the optimum conditions obtained. the salt sample that has been dried in an oven at 60 °C for 24 hours is taken as much as 10 g and put into a two-neck flask which already contains (g ratio PET: ml ethylene glycol 1: 3) is added 30 ml ethylene glycol and 0.06 g Na<sub>2</sub>CO<sub>3</sub> for 1.5 hours at a temperature of 196°C.

#### **Characterization of PET in sample**

FTIR was used to determine the functional groups of the sample by adding 0.99 g of KBr to 0.01 gram of the depolymerization powder resulted.

## **RESULTS**

#### **Determination of Optimum Condition of PET Glycolysis**

The glycolysis process is carried out by using ethylene glycol solvent and assisted by a sodium carbonate catalyst. Ethylene glycol can cause termination of the ester chain in the PET chain, while sodium carbonate catalyst was chosen because this catalyst is easily dissolved in ethylene glycol. The glycolysis reaction is carried out in a closed state so that no PET mass, ethylene glycol solvent, and sodium carbonate catalyst is lost. The glycolysis reaction was carried out for 1 hour starting from the optimum temperature (196°C) (López-fonseca *et al*, 2011).

Physically, the glycolysis process can be observed in three stages. First, before the optimum condition is reached, there is no change in PET and the mixture still shows a heterogeneous form, where the liquid phase is ethylene glycol and sodium carbonate and the solid phase is PET. Second, when the optimum has been reached, it appears that the PET has partially dissolved. Third, the final stage of the glycolysis reaction where the PET has completely dissolved, and the mixture is homogeneous. This homogeneous mixture shows that PET is starting to convert into oligomers and even monomers. The resulting glycolysis is a mixture of monomer, BHET, oligomer, ethylene glycol, catalyst and some unconverted PET (Amalia and Lukman, 2013). To obtain BHET solids, further refining is necessary. After the glycolysis reaction lasts for 1 hour, the three-neck flask containing the solution mixture is cooled in an ice bath. The cooling process is carried out immediately so that the reaction can stop. This is because the glycolysis reaction is reversible if the

reaction is not stopped, the product will return to the reactants (PET).

After the cooling process is complete, 70 ml of boiling distilled water is added to the reaction product in the form of a solid so that the solid dissolves (BHET is quite soluble in hot water). Because BHET monomer has a melting point of 109 °C [14] while BHET dimers have a melting point of 170 °C (Ghaemy and Mossaddegh, 2015). Then this process aims to separate BHET from oligomers (such as dimers and trimers). Furthermore, in a hot state, the results are filtered. From this first extraction, a filtrate containing BHET, ethylene glycol, and a small portion of oligomers dissolved in water was obtained and a residue in the form of PET that had not been converted.

The first extracted filtrate is heated until it is homogeneous and has a clear color. This process aims to re-dissolve the crystallized BHET as the filtrate temperature decreases and is filtered to ensure that no PET residue is mixed in the second filtrate. The second extracted filtrate was stored in the refrigerator for 16 hours at 5 °C for crystallization. The cooled crystals were filtered again to obtain BHET. From this separation, a yellowish clear filtrate is produced, i.e. ethylene glycol and dissolved catalyst and a precipitate in the form of solid BHET. The filtered precipitate was dried in an oven at 60 °C to evaporate the solvent and water remaining in the sediment. Then the solid is weighed to determine the mass of BHET produced.

The depolymerization process of PET plastic using the glycolysis method occurs because the nucleophilic substitution reaction on the hydroxyl group contained in ethylene glycol attacks the

carbonyl groups on the ester chain owned by PET. The carbonyl functional group is first activated by a cation on the catalyst. Sodium metal will bind to oxygen on the carbonyl to form a carbocation. This reaction is considered to be a complex formed by coordination between the carbonyl group on the ester and the metal sodium. The coordination that is formed decreases the electron density of the carbonyl group and opens the possibility of a nucleophilic attack from the hydroxyl group against the polarized carbon atoms, causing the PET polymer chain to break and produce BHET monomers.

The amount of sodium carbonate catalyst used in the glycolysis process can affect the yield of glycolysis. The following is the effect of the amount of sodium carbonate catalyst on the mass of glycolysis in Table 1 below.

**Table 1.** Effect of the amount of sodium carbonate catalyst on the BHET mass

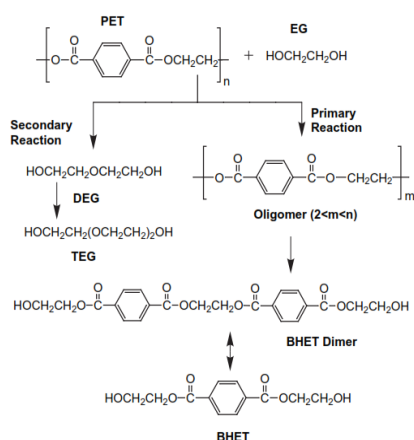
Na <sub>2</sub> CO <sub>3</sub> Mass (g)	BHET Mass (g)
0,02	4,7299
0,04	6,5523
0,06	7,2633
0,08	6,0788
0,1	4,9070

The yield percentage of the depolymerization process of PET by ethylene glycol using sodium carbonate as catalyst was calculated using the equation.

$$\% \text{ yield} = \frac{W_{\text{BHET}}}{W_{\text{PET}}} \times 100 \%$$

Where  $W_{\text{BHET}}$  is the weight of BHET resulting from the depolymerization process and  $W_{\text{PET}}$  is the initial weight of PET used. From the above equation, it can be seen the effect of the amount of Na<sub>2</sub>CO<sub>3</sub> catalyst on the yield of BHET with 10 g PET:30 ml ethylene glycol for 1 h at a temperature of 196°C as shown in Fig. 2.

Based on the figure, it can be seen that the highest BHET yield obtained was 72.63% when the addition of the mass of sodium carbonate catalyst was 0.06 gram. This result is supported by research (Amalia and Lukman, 2013) with the best yield obtained using Na<sub>2</sub>CO<sub>3</sub> is 64.11%. This is related to the selectivity of the catalyst, i.e. the ability of the catalyst to accelerate the reaction between several reactions so that the desired product can be obtained



**Fig. 1.** Glycolysis Reaction (Imran *et al*, 2010)

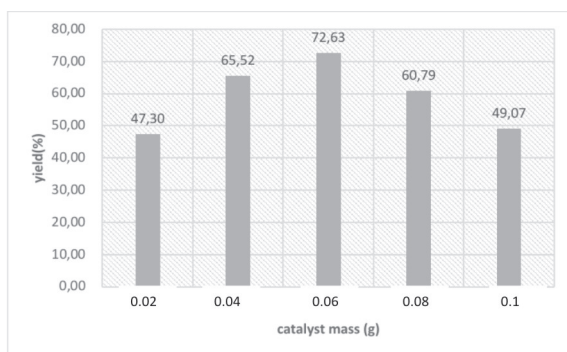


Fig. 2. Effect of catalyst on yield

with as few by-products as possible. With the addition of a catalyst 0.06 gram of the desired product reaches the highest amount with the products in the form of oligomers and dimers compared to other variations. If glycolysis is carried out by adding a sodium carbonate catalyst greater than 0.06 gram, i.e. the addition of 0.08 gram and 0.1 gram of catalyst, the results obtained tend to decrease. This decrease in yield is due to the decreased catalytic activity of sodium carbonate (Amalia and Lukman, 2013). PET glycolysis results were characterized using FT-IR at a wavelength of  $600 - 4000 \text{ cm}^{-1}$  to identify specific functional groups of BHET. BHET is a compound consisting of a hydroxy group (OH-) and a carbonyl group (C = O)

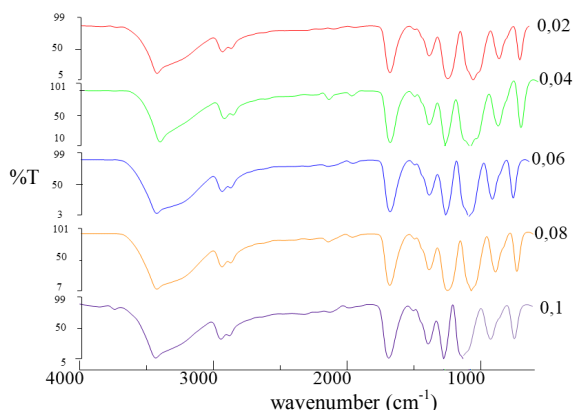


Fig. 3. FT-IR BHET spectra of various catalysts

shown in Fig. 3 below.

The carbonyl group on carboxylic acid (C=O) with a strong intensity is indicated by the presence of a peak in the number  $1690-1692 \text{ cm}^{-1}$ . At wavenumber  $3433-3435 \text{ cm}^{-1}$ , there is a moderate intensity for the -OH group on carboxylic acids. There is a peak with a strong intensity at numbers  $2945-2946 \text{ cm}^{-1}$  which indicates the stretching of the C-H bond ( $\text{sp}^3$ ). The C-O bond of the carboxylate group is shown in the wavenumber  $1260-1262 \text{ cm}^{-1}$  and  $1063-1066 \text{ cm}^{-1}$  with a strong intensity too. The spectra that have been described show similarities with the BHET spectra reported by Pingale and Shukla (2008), thus it can be proven that BHET has been formed.

The volume of ethylene glycol used in the glycolysis process can affect the yield of glycolysis. The following is the effect of the volume of ethylene glycol on the mass of glycolysis results.

The effect of EG volume on BHET yield is as shown in Fig. 4 below.

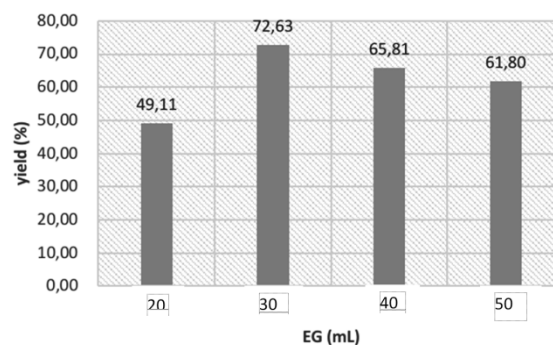


Fig.4. Effect of EG solvent on yield

Based on the graph, it can be seen that the optimum BHET yield is 72.63% when the PET: EG volume ratio is 10 g: 30 ml. This result is supported by research (Danarto *et al.*, 2012) which states that the highest BHET product was obtained at 1: 3 (g: ml) PET bottle: EG ratio (g: ml) with the results obtained at 69.72%. However, with the addition of EG volume that is greater than the ratio of 1: 3 does not make the results obtained will be more. This is

Table 2. FTIR absorption results of depolymerization products

Catalyst (g)	Wave number ( $\text{cm}^{-1}$ )					
	-OH	C-H	C=O	C-O	C-H	C-H
0.02	3434,78	2945,49	1690,49	1262,86	1066,53	719,33
0.04	3435,99	2945,93	1691,60	1262,89	1063,43	719,66
0.06	3432,40	2945,14	1691,01	1261,72	1064,39	716,48
0.08	3433,78	2946,69	1692,83	1262,71	1068,62	717,88
0.1	3433,24	2946,08	1691,62	1260,32	1063,02	719,47

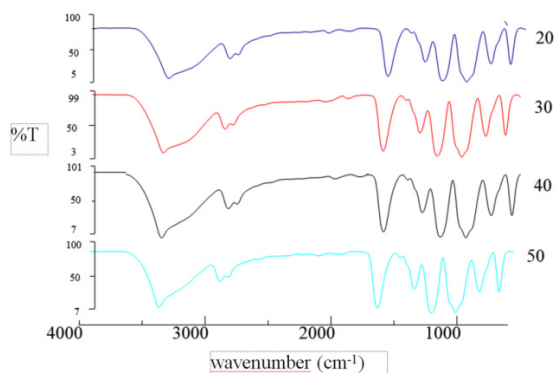
because the PET to be depolymerized has run out so it doesn't react with EG anymore.

**Table 3.** Effect of ethylene glycol volume on BHET mass

PET Mass: EG Volume (gm: ml)	BHET Mass (g)
10 : 20	4,9112
10 : 30	7,2633
10 : 40	6,5811
10 : 50	6,1803

In the FT-IR spectra, it is clear that the absorption peaks of several wavenumber are typical of BHET compounds. Among them, there is a peak with moderate intensity at the wavenumber 3432-3436  $\text{cm}^{-1}$  for the -OH group on carboxylic acids. The carbonyl group on carboxylic acid (C = O) with a strong intensity is indicated by the presence of a peak in the number 1690-1692  $\text{cm}^{-1}$ . At numbers 2945-2946  $\text{cm}^{-1}$  there is a peak with a strong intensity indicating the stretching of the C-H bond ( $\text{sp}^3$ ). The C-O bond of the carboxylate group is shown in the wavenumber 1260-1263  $\text{cm}^{-1}$ . and 1063-1066  $\text{cm}^{-1}$  with a strong intensity too. The spectra that have been described show similarities with the BHET spectra reported by Pingale and Shukla (2008), thus it can be proven that BHET has been formed.

Temperature has an important role in the PET glycolysis process because the high and low



**Fig. 5.** FT-IR Spectra of BHET at the EG variation

temperature will affect the termination of the PET polymer chain. The variation of reflux temperature was carried out in the temperature range of 166° C to 196° C using the optimum amount of ethylene glycol solvent obtained, which was 30 mL. The result show that there is an increase in the mass of BHET produced with increasing depolymerization temperature. This happens because at higher temperatures, the particles of PET have high kinetic energy so that the depolymerization reaction takes place faster. In addition, the use of ethylene glycol solvent which has a boiling point of 196 °C causes the depolymerization process to take place optimally at that temperature.

Seawater has the potential to be contaminated by microplastics because some of the plastic waste dumped into the river and ended up in the sea. Seawater samples were taken from three different beaches, i.e Padang Beach, Gajah Beach and, Tabing Beach. Seawater preparation uses a method that has been reported (Desforges *et al.*, 2014), i.e by filtering seawater. The residue on the filter paper was rinsed with seawater with added concentrated HCl and heated at 80-90 °C for 3 hours. This process aims to dissolve the organic material in seawater samples in concentrated HCl. Then, the residue obtained is filtered and dried in an oven at a temperature of 60 °C. The residue obtained is then refluxed at the optimum conditions as obtained from PET bottle glycolysis, i.e by adding 0.06 gm sodium carbonate catalyst and 30 ml ethylene glycol volume.

The second filtrate after being cooled for 16 hours in the refrigerator, there are no white crystals in the solution in the image, and the filter results there are also no white crystals which indicate BHET is present. This means that the seawater sample used does not contain PET so that during depolymerization no BHET crystals are formed. This occurs because PET has a density of 1.38  $\text{g}/\text{cm}^3$  while the density of seawater ranges from 1.026-1.028  $\text{g}/\text{cm}^3$ . With a density greater than seawater, PET tends to settle in the undersea or deep-sea sediments (Zhao *et al.*, 2018). PET microplastics were

**Table 4.** FTIR absorption results of depolymerization products

EG variation (mL)	Wave number ( $\text{cm}^{-1}$ )					
	-OH	C-H	C=O	C-O	C-O	C-H
20	3433,98	2946,7	1692,11	1260,14	1063,04	718,67
30	3432,43	2945,14	1691,01	1261,72	1064,39	716,48
40	436,37	2946,14	1690,78	1263,11	1067,61	720,01
50	3436,22	2946,99	1690,66	1262,79	1067,63	719.24

found in deep-sea sediments (35.2%) (Liu *et al.*, 2018) and (27.5%) (Zhang *et al.*, 2020). Meanwhile, microplastics that have a density less than that of seawater will float on the surface of sea water such as LDPE, HDPE, and PP (Tsang *et al.*, 2017), polyethylene, polypropylene, and polystyrene (Zhang *et al.*, 2017).

### CONCLUSION

Based on the results of the research that has been done, it can be concluded as follows: the optimum condition for depolymerizing PET using the glycolysis method is the addition of 0.06 gram of sodium carbonate catalyst with 30 mL of EG solvent with temperature of 196°C, the yield of BHET produced from glycolysis of PET bottles at the optimum condition was 72.63%, the absence of microplastic contamination of the PET type in seawater and salt samples in Padang City using the glycolysis method.

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