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Glycolysis Of Poly (Ethylene Terephthalate) (PET) Waste Under Conventional Convection-Conductive Glycolysis

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Abstract

Polyethylene terephthalate (PET) bottle wastes from disposable soft drink bottle were subjected to the depolymerization via glycolysis using excess of ethylene glycol. The reaction was carried out under conventional convection-conductive glycolysis in the presence of different class of metal catalyst as catalysts namely zinc acetate, sodium bicarbonate, zinc chloride and titanium (IV) oxide. The effect selection of catalyst and catalyst concentration (ratio catalyst:PET w/w%) on glycolysis depolymerization were investigated. After repeated crystallization, the glycolysis products were obtained with high conversion of 88%. It was subjected to the characterization by HPLC, TGA, DSC and FTIR analysis. It was found the glycolysis products consist mainly of bis 2-hydroxyethyl terephthalate (BHET) monomer with 75% recovery

Keywords: *Glycolysis, PET, depolymerization, glycolysis products, BHET monomer*

1. Introduction

Abundant amount of polyethylene terephthalate (PET) waste is becoming a serious problem due to the high consumption of various products such as soft-drink bottle, food container and etc. PET has excellent thermal and mechanical properties of PET make it more attractive to the industry [1,2]. The non-biodegradability of PET is the major obstacle to dispose this waste using conventional method such as landfilling and incineration due to the adverse environmental, economical aspect consideration and also the human health. Recycling is assumed one of the best approaches to solve the accumulation of PET waste problem taking into account ecological and economic factors. Moreover, recycling of PET lead to produce original feedstock of its monomer that can used for reproduce PET that can converted by mechanical methods into extruded or molded for better properties [2,3].

Several methods of chemical recycling of PET through different solvent (solvolysis) have been proposed since they lead to yield different types of final products. The chain cleavage of PET polyester is possible by used different reagent such as using amines such as ethanolamine in aminolysis of PET that lead to produce bis-2-hydroxyethylene terephthalateamide (BHETA) [4], also the use of alcohol such as methanol in alcoholysis that converts PET into dimethyl terephthalate (DMT) and ethylene glycol [5]. Hydrolysis of PET can be done under acidic and basic condition that involved the reverse of direct esterification reaction that yield the end-product of terephthalic acid (TPA) and ethylene glycol (EG). This process also gives along with the corrosion and pollution problems [6, 7].

Taking account the cost and energy consumption, glycolysis is one effective and preferable recycling method since it can be performed in the mild condition [3, 9]. Moreover, this process involved the degradation of PET molecular chain by glycols and is rapidly used in a commercial scale. Glycolysis commonly performed using ethylene glycol (EG), diethylene glycol, propylene glycol and dipropylene glycol in the presence of trans-esterification catalyst, commonly metal acetates where cleavage the ester linkage and substituted with diol or 2 hydroxyl terminals from glycol chain. [10,11]. Glycolysis can lead to the recovery of bis-2-hyroxyethyl terephthalate (BHET) from PET waste in the presence of excess ethylene glycol [4,11]. BHET has been used extensively in the synthesis of many polymeric materials such as unsaturated polyester resin, plasticizers, polymer concrete, polyurethane foam, textile dyes and softeners [11, 12]

Some previous study of glycolysis involve the kinetic of the process which reported the glycolysis rate of PET waste through different glycol in the presence of zinc acetate as trans-esterification catalyst was second order with respect to ethylene glycol concentration. Further investigation found that final product yield, BHET monomer and reaction rate depend on pressure, temperature, type and concentration of EG also concentration of catalyst. [11,13,14]. It has reported that the PET glycolysis reaction rate depend on the EG concentration with rate constant found inversely proportional to the reaction time and PET waste particle size as well as degree of depolymerization of PET waste inversely proportional to the particle size of PET waste [15].

Investigation by Chen [16], he found that the use of manganese acetate can optimized the production of BHET monomer in the glycolysis reaction that carried out at 190°C for 1.5 hours. Goje and Mishra [17] have reported that zinc acetate and cobalt acetate were displayed better catalytic effect than lead and manganese acetate in the glycolytic depolymerization of PET. Similar work is done by Lopez-Fonseca et al. [18] but in the glycolysis of post-consumer PET waste. They also found zinc acetate catalyst demonstrated the highest catalytic catalyst. Troev et al. [19] has introduced novel titanium (IV)-phosphate to replace conventional heavy metal catalyst and found that it possessed higher catalytic activity than zinc acetate when depolymerizing fibre-grade PET. Wang et al. [2] introduced ionic liquid catalyst of 1-butyl-3-methylimidazolium bromine *([bmim] Br*) that achieved high conversion into end-product compare to metal acetate catalyst.

Mansour and Ikladious [20] studied PET glycolysis utilizing different glycol solvent that found the glycolyzed product yielded consist of minor fraction of terephthalate oligoester. Meanwhile, Gouxi et al. [21] discussed effect of reaction time, EG/PET ratio and amount of acetate catalyst used and described that the best PET conversion occurs at the condition of 5:1 EG/PET mass ratio in 3h reaction by adding 1:100 catalyst:PET mass ratio. In this study, glycolysis was performed by using convection-conduction heating glycolysis in reflux method. The effect of using different classes of metal catalyst and catalyst concentration of ratio catalyst:PET are investigated. The glycolysis products obtained were characterized using several of analyses.

2. Experimental Works

2.1 Materials

PET waste from soft drink bottles was collected from local industry factory. The bottles was washed and cleaned and cut into small pieces of about 6 mm x 6mm. The chemical used were 98% analytical grade ethylene glycol and chromatography grade methanol for HPLC analysis (Thermo Fisher Ltd.), 99% zinc acetate dihydrate and 95% standard sample BHET (Sigma Aldrich)

2.2 Glycolysis

The experimental set up is made up of 500 ml three necked round bottom flask connected to a condenser. The ratio EG:PET 5:1 (w/w%) is subjected to the reaction vessel together with chosen catalyst of zinc acetate, sodium bicarbonate, zinc chloride and titanium (IV) oxide at temperature of 196°C under continuous nitrogen flow . After 8 hours the reaction is stopped and the solution is cooled to room temperature. An amount of boiling water then is added to eliminate excess EG left. The mixture then is filtered and the residue is collected, dried in 80°C oven, weighed and labelled as GP1. The filtrate is collected and cooled at 4°C for 24 hours to allow crystallization and glycolyzed products obtained labelled as GP2. Further crystallization is repeated and filtered and next crystallize product is labelled as GP3.The conversion of glycolyzed products from PET waste is defined by Eq. (1):

$$\%\text{ Product Conversion} = \frac{GP1 + GP2 + GP3 \times 100\%}{\text{Initial mass PET waste}} \quad (1)$$

where GP = glycolysis product

In order to obtained the composition of BHET monomer in the converted glycolysis products, the concentration of BHET monomer is obtained from HPLC quantitative analysis and is defined by Eq. (2)

$$\%\text{ BHET monomer} = \frac{I_{BHET}}{I_{GP}} \text{ X } 100 \qquad (2)$$

where I_{BHET} = Concentration of BHET
I_{GP} = Initial concentration of glycolysis product

2.3 Characterization

Thermal analysis on the glycolysis products is performed using Mettler Toledo thermogravimetric analyzer (TGA) model (TGA/STDA 851e). The mass loss of samples during temperature ranging from 25°C to 500°C is measured under nitrogen flow 10ml/min with heating rate of 10°C/min. Calorimetric of DSC scan is performed to the products obtained using Mettler Toledo Differential Scanning Calorimeter (DSC) model (DSC823e, Mettler Toledo, USA) under nitrogen atmosphere at flow rate 10ml/min in the temperature ranging from 25-250°C with increment temperature rate 10°C/min. IR Spectroscopy of Fourier Transform Infrared (FTIR) is done to identify the functional groups and chemical bond of samples using Perkin Elmer FTIR Spectrum 100 (USA). Chromatography analysis using HPLC was performed using Shimadzu HPLC model LC-20A Prominence HPLC and run by dissolving 4 mg samples in 50ml of mobile phase mixture of 70:30 v/v methanol and water at 254nm in flow rate of 1ml/min.

3. Results and Discussion

3.1 Effect selection of catalysts

The use of different catalyst may influence the glycolysis reaction. The effect of different classes of catalyst was investigated for their suitability to be used in the convection-conduction glycolysis. The chosen catalysts were zinc acetate (metal acetate), sodium bicarbonate (alkali catalyst), zinc chloride (metal chloride) and titanium (IV) oxide (metal oxide). In Fig.1, zinc acetate showed the highest catalytic effect judging from the highest conversion achieved greater than 88% yield. Sodium bicarbonate achieved 78% while zinc chloride and titanium (IV) oxide were at 68% and 56% respectively. Instead of highest product conversion, zinc acetate also exhibited highest total amount of BHET monomer. This is shown from the Table 1 where BHET recovery achieved 52% in GP1 with 80% and 92% in GP2 and GP3 respectively. This result has shown that zinc acetate exhibited the highest catalytic effect and become the most suitable catalyst in convection-conduction glycolysis of PET agreed with the previous works done by Baliga et al. [8] and Shukla and Kulkarni [22]. This finding confirm with other similar glycolysis of PET where zinc acetate exhibited the highest catalytic activity as compared to other metal acetate [8,22]. Sodium bicarbonate also a good catalyst for PET glycolysis depolymerization since it is a basic catalyst that possessed high solubility in ethylene glycol that can enhance the ability to penetrate, diffuse and swallow the PET structure. This will cause the reaction system become as one single homogenous liquid phase which has increase the glycolysis rate as compare to non-homogenous solid and liquid phase as explained by Pardal and Tersac [21] and Lopez et al. [18]. Furthermore, the use of this catalyst is safe because it is mild agreed as Shukla and Kalkurni [22] and eco-friendly with less harmful impact posed as opposed to the toxic zinc [18]. Zinc chloride also can be used for PET glycolysis depolymerization but its performance less superior compared to zinc acetate and sodium bicarbonate. This due to the unoptimzed condition applied in glycolysis since it required catalyst concentration of 0.5% w/w, with PET: EG ratio 1:14 under 8h reaction time [12]. Meanwhile, titanium (IV) oxide has the lowest catalytic activity compared to the other catalysts and this could be this catalyst has well in polymerization of PET as reported Shah *et al.*[23].

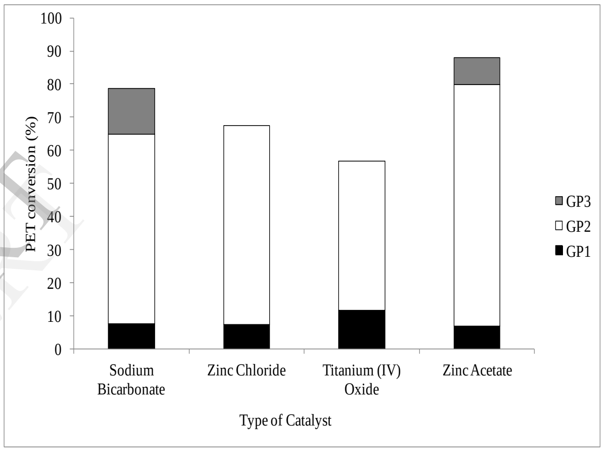

Fig.1: Effect selection of catalyst in convection-conduction glycolysis

Table 1: Concentration of BHET monomer on effect of selection catalysts

Type of catalysts	Glycolysis product, GP1		Glycolysis product, GP2		Glycolysis product, GP3		Average BHET recovery (%)
	BHET (ppm)	BHET (%)	BHET (ppm)	BHET (%)	BHET (ppm)	BHET (%)	
Sodium Bicarbonate	12	15	16	20	29	36	24.0
Zinc Chloride	22	28	38	47	-	-	25.0
Zinc Acetate	42	52	64	80	73	92	75.0
Titanium (IV) Oxide	8	11	13	16	-	-	9.0

3.2 Effect of catalyst concentration (ratio catalyst: PET w/w %)

Fig. 2 exhibited the effect of adding catalyst at different concentration influence the product conversion in glycolysis. The product conversion increased by increasing the ratio with maximum conversion reached in 88% after loading 1.5% (w/w) zinc acetate. Further increasing the ratio to 2.0% (w/w) catalyst the conversion showed a slightly decrement less than 80%. This trend result is similar occurred in the conversion of BHET monomer in Table 2, where the BHET concentration first appeared to increase with increasing the amount of catalyst, and then the BHET recovery appeared to decrease when the amount catalyst loaded is more than ratio 1.5% (w/w). Under the optimum of catalyst concentration obtained, when the load of zinc acetate catalyst is 1.5% (w/w), the PET conversion has reached into maximum glycolysis products of 88% and has recovered BHET monomer at 75%.

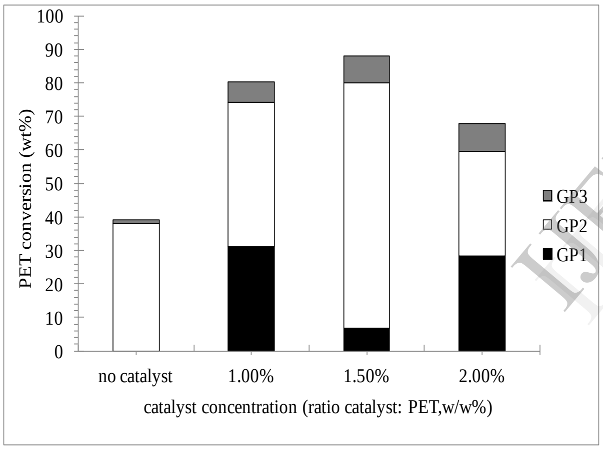

Fig. 2: Effect of catalyst concentration (ratio catalyst:PET, w/w%)

Table 2: Concentration of BHET monomer on effect of catalyst concentration

Catalyst concentration Catalyst:PET (w/w%)	Glycolysis product, GP1		Glycolysis product, GP2		Glycolysis product, GP3		Average BHET recovery (%)
	BHET (ppm)	BHET (%)	BHET (ppm)	BHET (%)	BHET (ppm)	BHET (%)	
No catalyst added	4	5	16	20	25	32	19.0
1.0%	26	32	35	43	62	78	51.0
1.5%	41	52	64	80	73	92	75.0
2.0%	18	23	24	30	34	43	32.0

3.3 Characterization of product

3.3.1 Chromatography analysis (High Performance Liquid Chromatography-HPLC)

Identification and separation of BHET monomer in the product recovery from glycolysis was investigated using HPLC qualitative analysis. The sample solution of 80 ppm concentration was prepared by dissolving about 4 mg glycolysis products in 50 ml of methanol/water (70/30 v/v) mixture. A typical HPLC chromatogram of control BHET monomer is exhibited in Fig. 3(a) It is shows one sharpest peak at the retention time of 3.375 min which indicated the complete separation of BHET monomer. The small broad peak at the retention time of about 4.50 min suggested the presence of small amount of BHET dimer. The chromatogram of one selected glycolysis product (GP2) is shown in Fig. 3(b). This chromatogram exhibited that GP2 possessed similar chromatogram as BHET control with one dominant peak at the retention time about 3.380 min together a small broad peak in time about 4.50 min. This is shown that GP2 composed mainly of BHET monomer with small composition of its dimer.

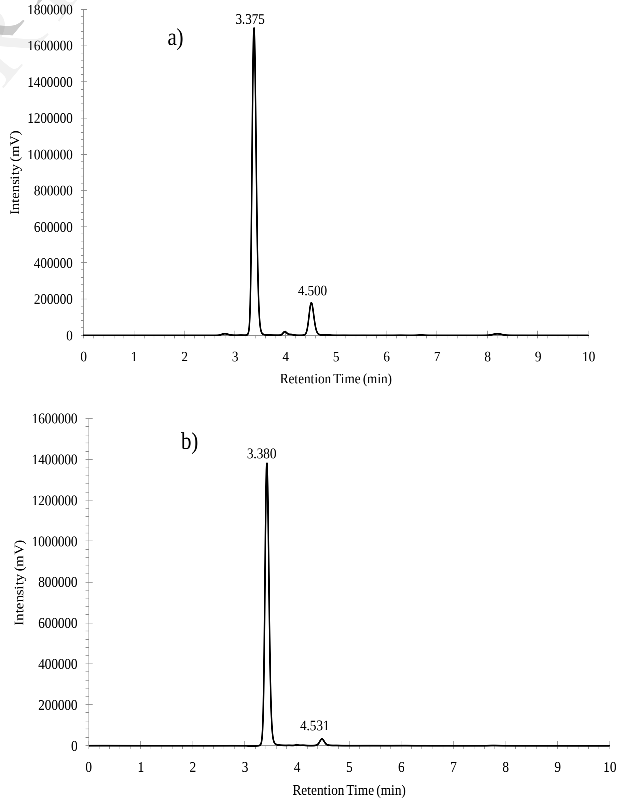

Fig.3: Chromatogram of BHET control (a) and GP2 from glycolysis (b)

3.3.2 Thermal Analysis

Four TGA curves of glycolysis products and control BHET monomer are shown in Fig 4. Control BHET exhibited its decomposition region in the two different temperatures. The first decomposition is occurred in range temperature 190-220°C and second decomposition occurred at 380-400°C. The TGA curves for glycolysis products also exhibited similar trend decomposition as BHET control. The samples were thermally stable until 190°C and started to decompose in onset temperature of 198°C. Rapid mass loss has occurred in first decomposition step in range temperature 200-220°C with 25% of component has decomposed. The second mass loss occurred in range 400-420°C with higher amount of 60% component has decomposed. The first mass loss that occurred at 220-220°C was due to the thermal degradation of BHET monomer while in the second mass loss occurred at 400-420°C is attributed to the thermal degradation of high chain oligomers or PET which produced by the BHET thermal polymerization during the thermogravimetric analysis. This result has agreed with the previous research by Wang *et al.*[2] and Chen [16] in determine the thermal properties of glycolysis products. Furthermore, it also indicated that during TGA analysis, there are thermal polymerization has occurred when short chain products of BHET monomer, and its dimer has converted into trimer or oligomer and oligomer to PET [16].

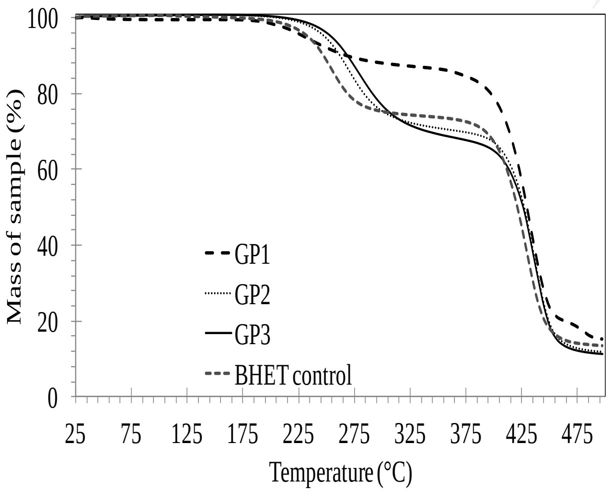

Fig. 4: TGA curves of control BHET monomer and glycolysis products from conductive-conduction glycolysis

Calorimetric analysis has done using DSC instruments and the DSC thermograms of the BHET control and glycolysis products are shown in Fig 5. The control BHET monomer exhibited onset melting occurred at 108°C and peaked at 110°C. GP1, GP2 and GP3 exhibited similar onset of melting peak ~108°C, but shifted slightly at the range of 109-111°C. The endothermic peaks recordered in all samples are similar with BHET monomer since it has agreed that in the range of 108-111°C. This corresponds well to the peak of BHET monomer as obtained by other researchers Guclu [24] and Shukla [14]. GP1 exhibited a broad hump in the range 130° - 150°C suggested the presence of an amount of BHET dimer. The difference in the amount of heat flow indicates the amount of energy consumed for phase transition of the glycolysis products. This is the amount of heat energy required to change the physical state of glycolysis products from it solid state to molten state or liquid state. The sharper endothermic peak and the higher heat flow mean the higher the purity of BHET monomer. Result showed GP1 has the lowest BHET monomer with 10mW heat flow. This also contributed by the presence of BHET dimer, as indicated as the broad peak at 130-150°C that causing reduction in monomer purity. There is no much difference between heat flow of BHET control and GP2, at 40mW and 45 mW respectively. Thus it suggested that there is no significant different between GP2 and BHET control in term of BHET purity.

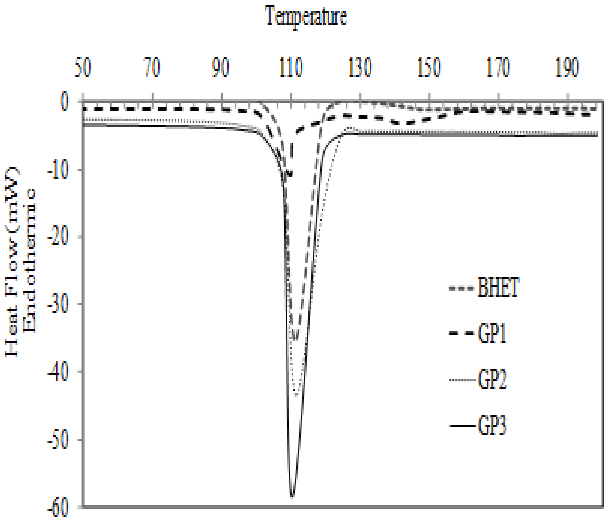

Fig. 5: DSC curves of control BHET monomer and glycolysis products from conductive-conduction glycolysis

GP3 has higher heat flow at 60mW suggests exhibition of the highest on BHET purity among other sample and its purity has exceeded the purity of control BHET monomer.

3.4.2 IR Spectroscopy analysis (Fourier Transform Infra Red -FTIR)

The FTIR spectrum of the BHET control and glycolysis products is shown in Fig. 6. It can be seen all of the samples exhibited similar spectrum as BHET control. Table 3 summarized the IR absorption frequency and chemical bonds that have identified. The absorption of 3440-3500 cm^{-1} indicated the presence of hydroxyl (-OH) alcoholic group. At 2850-3000cm^{-1} shown the presence of C-H stretch band, C=O at 1690-1720cm^{-1}, C-O-C at 1100cm^{-1} and aromatic ring at 675-900cm^{-1} similar as reported Abu Bakar [25]. Other –OH bond also identified at 1135cm^{-1} and C-H aromatic bonds at 1456-1504^{-1} agreed with previous report by Pingale et al. [12] while C-O ester asymmetric vibration at 1253-1274cm^{-1} and C-O ester symmetric vibration at 1072-1135cm^{-1} also detected similar as reported Moral et al.[26]. The FTIR spectrum has described that the molecular of each sample do not defer much from each other. However, the different sharpness of the peak can explained the different purity of sample. Thus, due to a much sharper peak is shown in GP3 rather than GP2 and GP1, it suggested GP3 achieved the highest purity of sample.

Table 3: IR absorption and chemical bonds of glycolysis products from convection-conduction glycolysis

Wavelength number (cm^{-1})	Functional group/ Chemical bond
3440-3500	O-H alcoholic group
2850-3000	C-H stretch (bend)
1690-1720	C=O
1456-1504	C-H aromatic bond
1253-1274	C-O ester asymmetric vibration
1135	O-H bond
1100	C-O-C
1072-1135	C-O ester symmetric vibration
675-900	Aromatic ring

4. Conclusion

Recycling of PET waste via convection-conduction glycolysis was successfully performed using different class of metal catalyst. It demonstrated that zinc acetate could behave as the most efficient catalyst for the PET depolymerization in ethylene glycol. A great in PET conversion and BHET yield was observed when the glycolsis reaction carried out in optimum condition of 5:1 solvent concentration (ratio PET:EG) with catalyst concentration 1:5% (ratio catalyst:PET) with the conversion of PET and BHET recovery has reached 88% and 75% respectively.

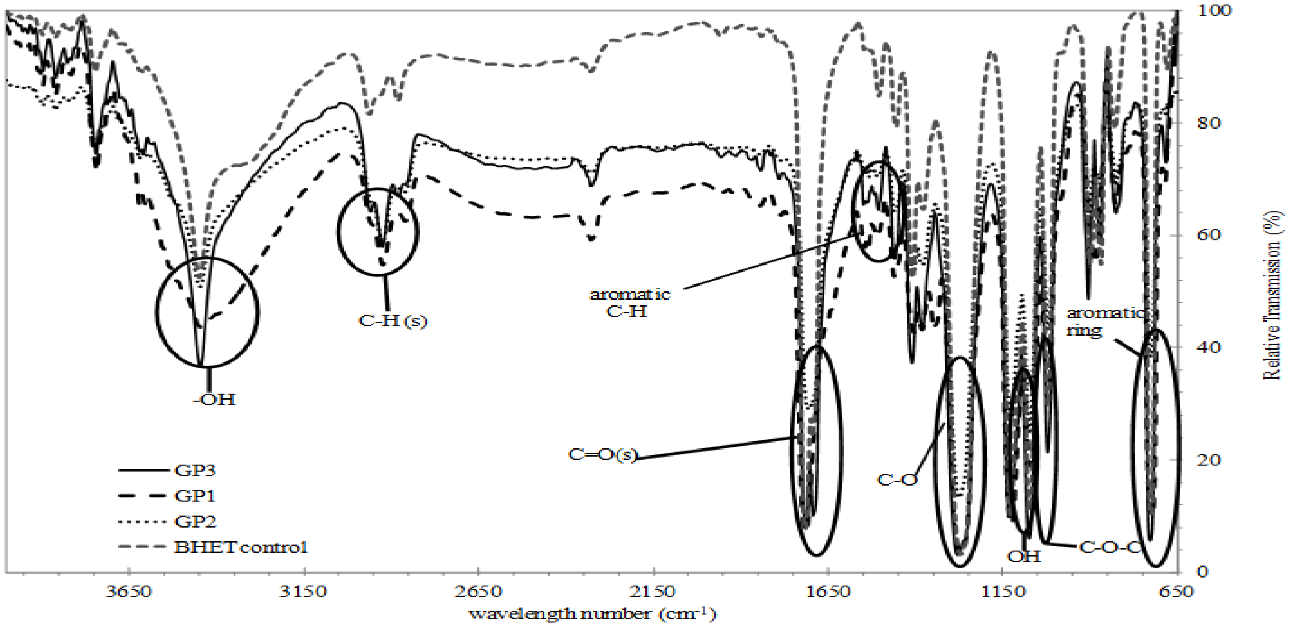

Fig. 6: FTIR spectrum of control BHET and glycolysis products of convection-conduction glycolysis

Various analysis on glycolysis products indicated that the samples composed mainly of BHET monomer. Chromatogram analysis via HPLC proves the separation and identification of BHET monomer in glycolysis products with elution occurred in similar retention time. Thermal analysis by TGA has confirmed the mass loss and decomposition properties of glycolysis products similar as BHET monomer with two step degradation occured. DSC analysis further confirms the melting point of BHET as reported in the literature. Furthermore, the structure of BHET monomer was confirmed by FTIR spectroscopy that indicating characteristic of chemical bond in the structure.

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