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Research Article

Time, Temperature and Amount of Distilled Water Effects on the Purity and Yield of Bis(2-hydroxyethyl) Terephthalate Purification System

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Abstract

Polyethylene terephthalate (PET) bottle is one of the common plastic wastes existed in the municipal solid waste in Malaysia. One alternative to solve the abundant of PET wastes is chemical recycling of the wastes to produce a value added product. This technology not only can decrease the PET wastes in landfill sites but also can produce many useful recycled PET products. Bis(2-hydroxyethyl) terephthalate (BHET) obtained from glycolysis reaction of PET waste was purified using crystallization process. The hot distilled water was added to glycolysis product followed by cooling and filtration to extract BHET in white solid form from the product. The effect of three operating conditions namely crystallization time, crystallization temperatures and amount of distilled water used to the yield of crystallization process were investigated. The purity of crystallization products were analyzed using HPLC and DSC. The optimum conditions of 3 hours crystallization time, 2 °C crystallization temperature and 5:1 mass ratio of distilled water used to glycolyze solid gave the highest yield and purity of the crystallization process.

Keywords: Optimum conditions; BHET; Crystallization Process; PET Waste; Yield

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1. Introduction

Polyethylene terephthalate (PET) is a common synthetic polymer available in the market, produced via two-stage step-growth polycondensation from ethylene glycol (EG) and terephthalic acid (TPA). The first stage is the esterification reaction between EG and TPA to form bis(2-hydroxyethyl) terephthalate (BHET) monomer and oligomer followed by polycondensation reaction to form PET polymer [1].

Due to its excellent strength characteristics, good gas barrier, transparency, chemical and thermal stability [2-3], PET is widely used in manufacturing of various types of products such as bottles, fibers and films. Pira International forecast the world consumption for PET packaging will increase to almost 19.1 million tons by 2017 with the market will grow 5.2% annually [4]. Growing consumption of PET leads to a continuous increase in the PET waste generation, and the main challenge is to keep pace in the waste management. PET wastes are not naturally degraded [5], therefore recycling of PET wastes can be used as one alternative to solve the abundance of PET

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wastes. In addition, PET recycling offers a good potential to minimize the dependency on fossil fuel as a raw material in PET production. Thus, recycled PET has a prospective to substitute virgin PET produced from refined fossil fuels [6]. On top of that, the energy required to produce recycled plastics is less than the production of virgin polymers from fossil fuels [7].

PET can be recycled via mechanical or chemical means. Mechanical recycling of PET wastes includes sorting, washing, drying and melt processing [8]. Sorting, washing and drying processes are to remove the contaminant found in PET wastes. On the other hand, chemical recycling of PET wastes comprises depolymerisation reaction, decontamination and re-polycondensation process. The purpose of depolymerization is to depolymerize PET to form BHET monomer. Available PET depolymerization method to date are hydrolysis [9], glycolysis [10], methanolysis [11], aminolysis [12] and ammonolysis [13]. The BHET produced from depolymerization reaction can be purified via crystallization, deionization, decoloring, distillation and many others. After decontamination process, purified BHET will be polymerized to form PET resin followed by PET product. Chemical recycling is preferable due to the requirement of high purity of recycled PET product.

Glycolysis product contains BHET, catalyst, solvent, unreacted PET, oligomers, impurities and many others. Crystallization process is used to extract the BHET from glycolysis product by using distilled water with agitation, boiling, chilling and filtration process [14, 15]. Pilati *et al*. [16] conducted a controlled crystallization process. In this method, EG solvent was removed from the glycolysis product, followed by an addition of hot water at 65 to 75 °C to dissolve and separate BHET from the oligomer found in the residue. The hot aqueous solution was cooled to 5 to 15 °C to extract the BHET. At the end of crystallization process, a hot water at 70 to 80 °C was re-added to the BHET obtained followed by a cooling process to 5 to 15 °C for further crystallizing the BHET.

The present work deals with the purification process performed in the chemical recycling of PET waste. Glycolysis product was purified using crystallization process to obtain BHET in the white solid form. In this work, a series of experiments were conducted with different crystallization time, crystallization temperature and different amount of distilled water used to investigate the optimum condition of crystallization process.

2. Materials and Methods

2.1. Material Preparation

PET flakes, post-consumer shredded PET bottles, were obtained from a local collector, Emanz Technologies (M) Sdn. Bhd. Both ethylene glycol (EG) and zinc acetate dehydrate analytical grade were purchased from Merck KGaA and Sigma Aldrich Co. respectively. HPLC grade methanol was supplied by Fisher Chemical Sdn. Bhd. Feedstock for the crystallization process was prepared using glycolysis reaction. Glycolysis was used to depolymerize PET polymer to BHET monomer. Figure 1 displays the reaction of glycolysis process. The PET flakes was treated with EG at a mass ratio of 1:5 (PET:EG) and 1.5% zinc acetate based on weight of PET flakes as catalyst under reflux in the presence of nitrogen gas. The reaction was carried out at 196 °C for 8 hours. The schematic diagram for the set-up of glycolysis proc-

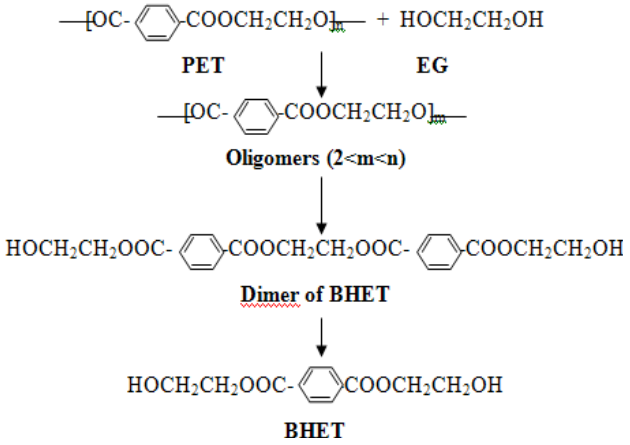

Figure 1. Glycolysis reaction [14]

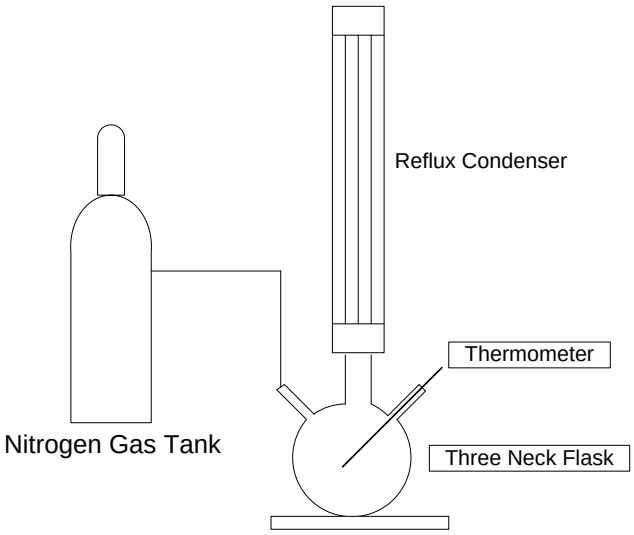

Figure 2. Schematic diagram of the glycolysis process

ess is shown in Figure 2.

2.2. Crystallization Process

Glycolysis product was cooled at 5 °C for 24 hours. White solid formed in the glycolysis product was filtered from the liquid which is the unreacted EG. The white solid obtained is named as glycolyzed solid (GS). The GS was purified using crystallization process. Hot distilled water (around 75 °C; dH_2O) was added to GS at a mass ratio of 1:1 to 1:7 (GS:dH_2O) to form homogeneous solution. The solution was cooled to 1-30 °C for 0.5 to 5 hours of crystallization time. At the end of crystallization process, white solid was formed and filtered from the distilled water. The white solid was dried in an oven at 70 °C and weighed. The white solid obtained from the crystallization process is named as crystallization product (CP). CP was subjected to different characterization techniques. The yield of solid product obtained from the crystallization process was calculated based on Equation (1).

$$\%\ of\ CP\ recovered = \frac{Mass\ of\ CP\ obtained}{Mass\ of\ GS} x100\% \quad (1)$$

2.3. Product Analysis

Differential Scanning Calorimeter (DSC) was used to examine the thermal properties of the product. The DSC thermograms were obtained using DSC 823/500 (Mettler-Toledo (M) Sdn. Bhd.) with 5 mg samples was heated at 10 °C/min in the range of 25 to 300 °C [5] under nitrogen gas flow of 10 cm^3/min.

High Performance Liquid Chromatography (HPLC) (Shimadzu SPD-20A DGU-20A5) was used to identify the composition of BHET in the crystallization product. The HPLC was equipped with a high resolution C18 column with 25 cm long and 4.6 mm diameter and UV detector set at 254 nm. HPLC analysis was performed using a mixture of methanol/water at volume fraction of 70/30 as the mobile phase at a flow rate of 1 ml/min [5]. The injection volume was 20 µl and the column temperature was set at 30 °C. HPLC chromatogram was analyzed using external standard method [17] to calculate the percentage of BHET containing in the CP. Four sets of methanol/water mixture containing 1.1 mg, 2.2 mg, 4 mg and 5.2 mg of BHET were analyzed using HPLC. Example of HPLC chromatogram of methanol/water mixture containing 1.1 mg of BHET is displayed in the appendix (Figure A1). The peak area data displayed in HPLC result was recorded. The concentration of BHET was calculated in Equation (2).

$$BHET\ Conc. = \frac{Mass\ of\ BHET, mg}{Vol.of\ methanol/water\ mixture, ml} \quad (2)$$

Table 1 shows the peak area and retention time of BHET extracted from the HPLC chromatogram of the four sets of methanol/water mixture and BHET concentration. This data was used in performing the calibration curve between the peak areas as a function of the BHET concentration.

Figure 3 displays the calibration curve of the peak area as a function of the BHET concentration. The concentration of BHET in the CP was calculated based on the peak area shown in the HPLC data according to the calibration curve. Hence, the composition of BHET in the CP was calculated in Equation (3).

$$BHET\ Comp.in\ CP, \% = \frac{Conc.\ of\ BHET}{Conc.of\ CP} x100\% \quad (3)$$

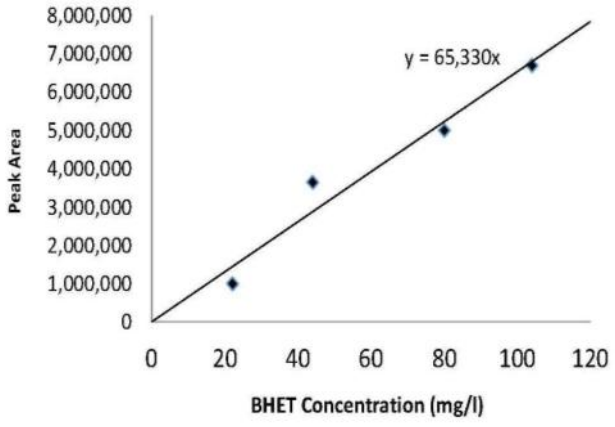

Figure 3. HPLC calibration curve

Table 1. Raw data of HPLC result and BHET concentration

Mass of BHET, mg	Volume of methanol/water mixture, ml	BHET concentration, mg/l	Peak Area	Retention Time of BHET
1.1	50	22	1,013,120	3.382
2.2	50	44	3,659,300	3.386
4	50	80	5,015,148	3.387
5.2	50	104	6,714,433	3.375

3. Results and Discussion

3.1. Glycolysis Product

10 g of PET flakes was depolymerized using 45 ml of EG and 0.15 g of zinc acetate dehydrate as catalyst via glycolysis reaction producing 55.47 g of glycolysis product. Cooling and filtration of the glycolysis product was performed to obtain the unreacted EG (42.85 g) and glycolyzed solid (GS, 12.62 g).

Glycolysis product was analyzed using DSC and HPLC to investigate the composition of BHET. Figure 4 shows the DSC thermogram of the glycolysis product. The melting point of BHET is commonly found in the range of 106 to 110 °C [5, 18]. A broad endothermic peak was observed in the DSC thermogram of glycolysis product in the region of 80 to 180 °C indicating an impure substance [16] consisting of BHET in the product. Another broad endothermic peak was observed in the region of 180 to 250 °C indicating glycolysis product containing oligomer.

HPLC chromatogram of the glycolysis product as displayed in the appendix (Figure A2) further proves the product contains BHET due to a dominant peak with the peak area of 1,084,934 at retention time of 3.385 minutes. By using the calibration curve (Figure 3), the BHET concentration is 16.607 mg/l. Using Equation 3, the calculated percentage of BHET in the glycolysis product is 18.87 % BHET. Hence, the estimated glycolysis product contains 19 % BHET, 77 % EG and the remaining is oligomer and impurities.

3.2. Effect of Time on Crystallization Product

The crystallization process was used in decontaminating the GS obtained from glycolysis reaction. The crystallization process was carried out at 9 °C crystallization temperature, using 1:7 for GS:dH_2O and crystallized at various crystallization times of 30 to 300 minutes. The percentage of CP recovered from GS at different crystallization times is displayed in Figure 5. The percentage of CP recovered at varying crystallization times of 30 to 300 minutes was 5.3 to 28.4 %. The result shows that higher crystallization time enables more BHET to crystallize and separate from the distilled water.

As shown in Figure 5, the CP recovered at 5 hours (300 minutes) crystallization time was 28.40% whereas at 3 hours (180 minutes) crystallization time, 27.13% CP was recovered, which only 1.3 % less than the 5 hours. In other words, by extending additional 2 hours (120 minutes) of crystallization time, the CP recovered was only increased by 1.3%. Reducing the time of the process means lesser power usage during the crystallization process. As a result, 3 hours crystallization period was chosen as the best crystallization time due to economic reason.

Figure 6 presents DSC thermogram of CP obtained from crystallization process at 3 hours crystallization time. The thermogram shows a sharp endothermic peak at 110 °C indicating the CP obtained consists of high purity of BHET with minimal impurities.

The dominant peak shown in the HPLC chromatogram (displayed in the appendix; Figure A3) at the retention time of 3.387 minutes shows that the CP contains BHET monomer. Based on the peak area of BHET (4,535,614) and using the calibration curve (Figure 3), 69.426 mg/l BHET concentration was calculated. The estimated purity of the BHET in the CP obtained at 3 hours crystallization time was increased to 80.73 % using Equation 3 with the

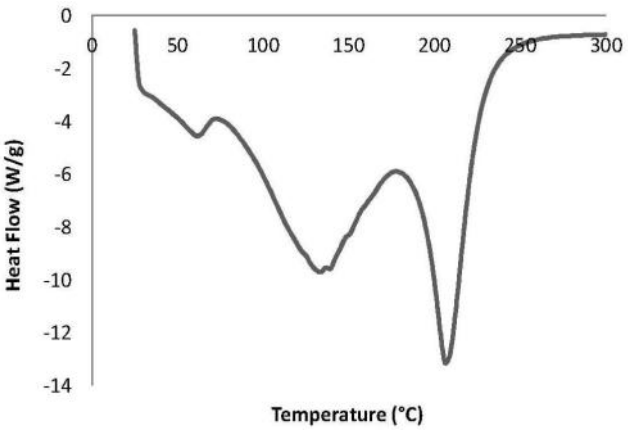

Figure 4. DSC thermogram of glycolysis product

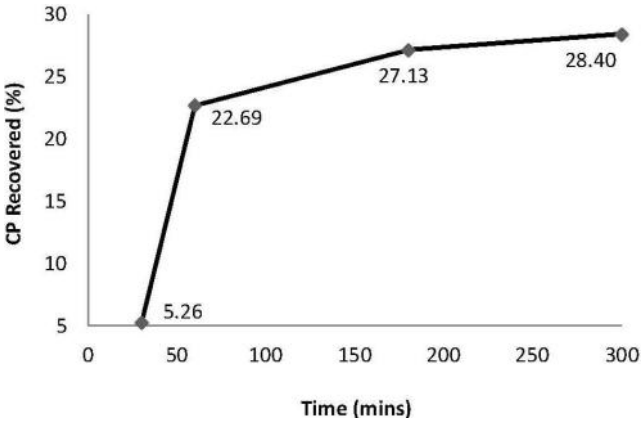

Figure 5. Percentage of CP recovered from crystallization process carried out at various crystallization time

remaining is oligomers (19.27%).

3.3. Effect of Temperature on Crystallization Product

The crystallization process was further investigated at various operating temperatures ranging from 1 to 30 ºC, while time and mass ratio of distilled water to GS were kept constant at 3 hours and 7:1 respectively. The percentage of CP recovered was 2.47 % to 32.94 % at various crystallization temperatures as illustrated in Figure 7. Lower percentage of CP recovered was observed at higher crystallization temperature. The main component in CP is BHET, thus the trend of CP recovered at higher temperature should be opposite to the BHET solubility which is increase as temperature increased [16]. This mean that, at higher temperature, more BHET would dissolve in the distilled water thus reducing the BHET crystallized from the distilled water.

Figure 8 shows DSC results of the CP obtained at 2 °C crystallization temperature. The presence of BHET in the CP was confirmed by a sharp endothermic peak at 110 °C. DSC thermogram also shows that the product crystallized at 2 °C exhibited one broad peaks at 170 to 270 °C indicating the existence of oligomers in the product.

The chosen optimum temperature in the crystallization of glycolysis product to extract BHET was 2 ºC due to maximum CP obtained (32.94%; Figure 7). A dominant peak at retention time of 3.391 shown in the HPLC chromatogram (appendix; Figure A4), proves that the CP contains BHET. With the peak area of BHET (6,770,895), 103.641 mg/l BHET concentration was obtained using the calibration curve. As a result, higher purity of BHET with 90.91% BHET was further obtained from the CP crystallized at 2 °C (oligomers is 9.09%).

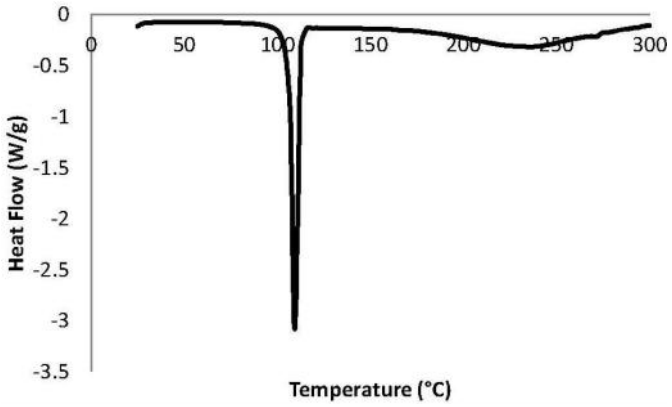

Figure 6. DSC thermogram of CP obtained at 3 h crystallization time

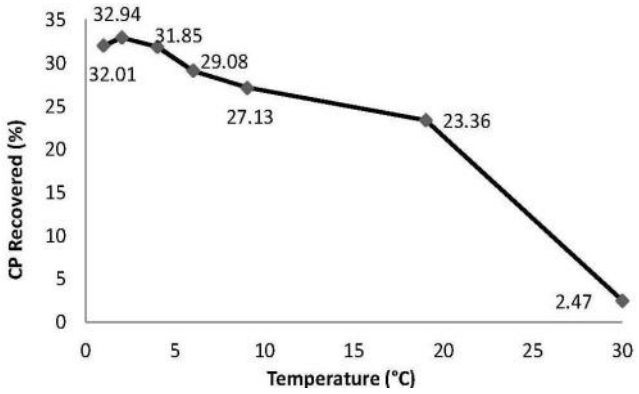

Figure 7. Percentage of CP recovered from crystallization process carried out at various crystallization temperature

3.4. Effect of Ratio of Distilled Water Used to Glycolyzed Solid (GS) on Crystallization Product

The crystallization process at 2 °C with 3 hours crystallization time was further investigated by varying the mass ratio of distilled water used to GS (1:1 to 7:1). The percentage of CP recovered at various mass ratio of distilled water to GS was in the range of 31.2 % to 39.4 % as portrayed in Figure 9.

Figure 10 illustrates the DSC thermogram of the CP obtained from the crystallization process using 5:1 mass ratio of distilled water to GS. A sharp endothermic peak was observed at 110 °C confirming the presence of BHET. Another peak was also observed at temperature range between 180 to 240 °C indicating the CP crystallized at 2 °C with 3 hours of crystallization time and 5:1 (dH_2O:GS) mass ratio contained unwanted oligomer.

The selected optimum mass of distilled water used in crystallization of GS was five times

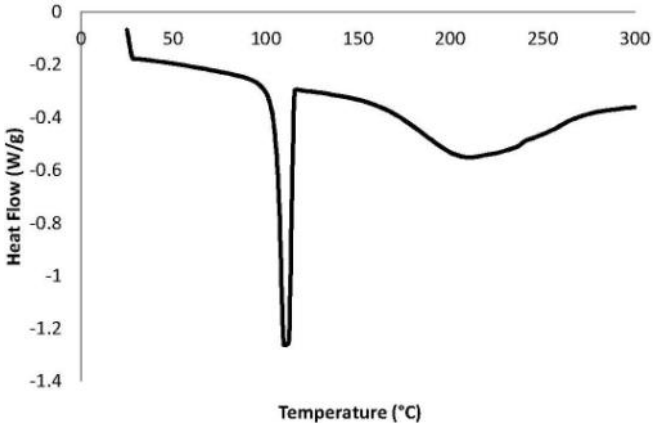

Figure 8. DSC thermogram of CP obtained at 2 °C crystallization temperature

to the mass of GS. At 5:1 (dH_2O:GS) mass ratio, maximum CP recovered was obtained at 39.44 %. A dominant peak at retention time of 3.389 minutes as shown in the HPLC chromatogram (appendix; Figure A5) indicates the presence of BHET in the recovered CP. The peak area of BHET is 6,441,681; and therefore 98.602 mg/l BHET concentration was obtained using the calibration curve. Based on the HPLC and the calibration curve analysis, higher purity of BHET with 93.02 % was further recovered from the CP obtained at optimum condition (with only less than 7% oligomers).

3.5. Comparison between Glycolyzed Solid and Crystallization Product

Table 2 shows the composition of glycolyzed solid (GS) and crystallization product (CP). The crystallization process at 2 °C by using distilled water to GS mass ratio of 5:1 and crystallized for 3 hours was able to increase the purity of BHET in the glycolysis product from 19 % to 93 %. As compared between the DSC thermogram of CP (Figure 10) obtained at optimum condition and glycolysis product before the crystallization process (Figure 4), the CP contains higher purity of BHET due to a sharp endothermic peak at 110 °C revealing the melting point of BHET. Therefore, crystallization at optimum condition was able to remove impurities in the glycolysis product yielding high purity of BHET.

Table **2.** The composition of glycolyzed solid and crystallization product

Components	Composition, %	
	Glycolyzed Solid	Crystallization Product
BHET	19	93.02
Oligomer and impurities	4	6.98
EG	77	0

4. Conclusion

Crystallization process was used in the purification of glycolysis product to recover and purify BHET monomer. The influence of three operating parameters namely crystallization time, crystallization temperature and ratio of distilled water used to GS were investigated in order to obtain the optimum condition of the crystallization process. 2 °C crystallization temperature, 3 hours crystallization time and 5:1 mass ratio of distilled water to GS was chosen as the optimum operating parameters yielding CP with 93 % BHET. Higher crystallization temperature reduced the percentage of CP recovered from GS whereas the percentage of CP recovered increased as the crystallization time increased. As a conclusion, crystallization process can be used to purify the glycolysis product produced during chemical recycling of PET waste.

Acknowledgement

The authors would like to express gratitude to Mr. Amir Syarriffudeen for the assistance during experimental and analysis work and to the Universiti Putra Malaysia in particular the Department of Chemical and Environmental Engineering for facility and funding.

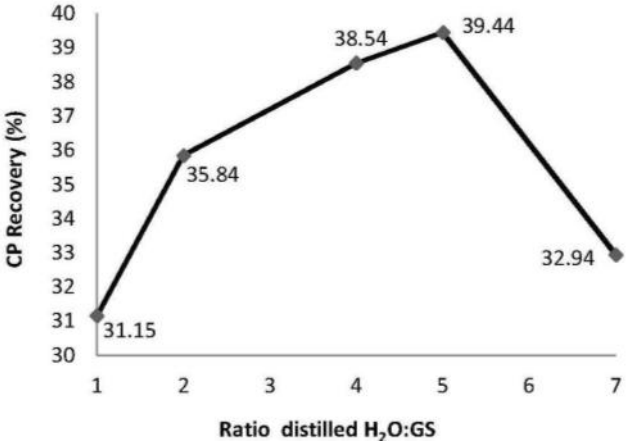

Figure 9. Percentage of CP recovered from crystallization process carried out at various mass ratio of distilled water to GS

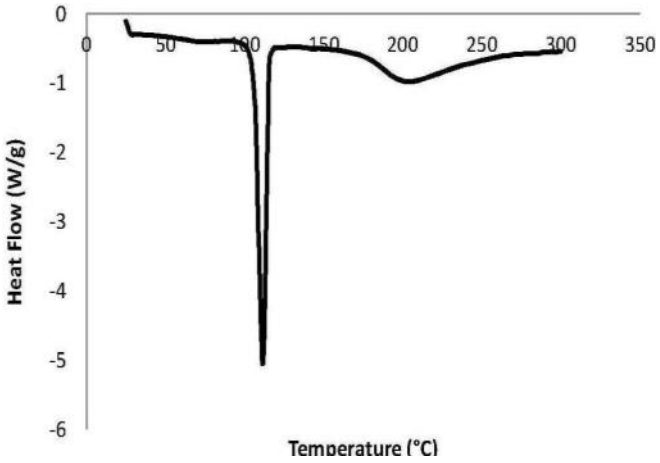

Figure 10. DSC thermogram of CP obtained using mass ratio 5:1 of distilled water to GS

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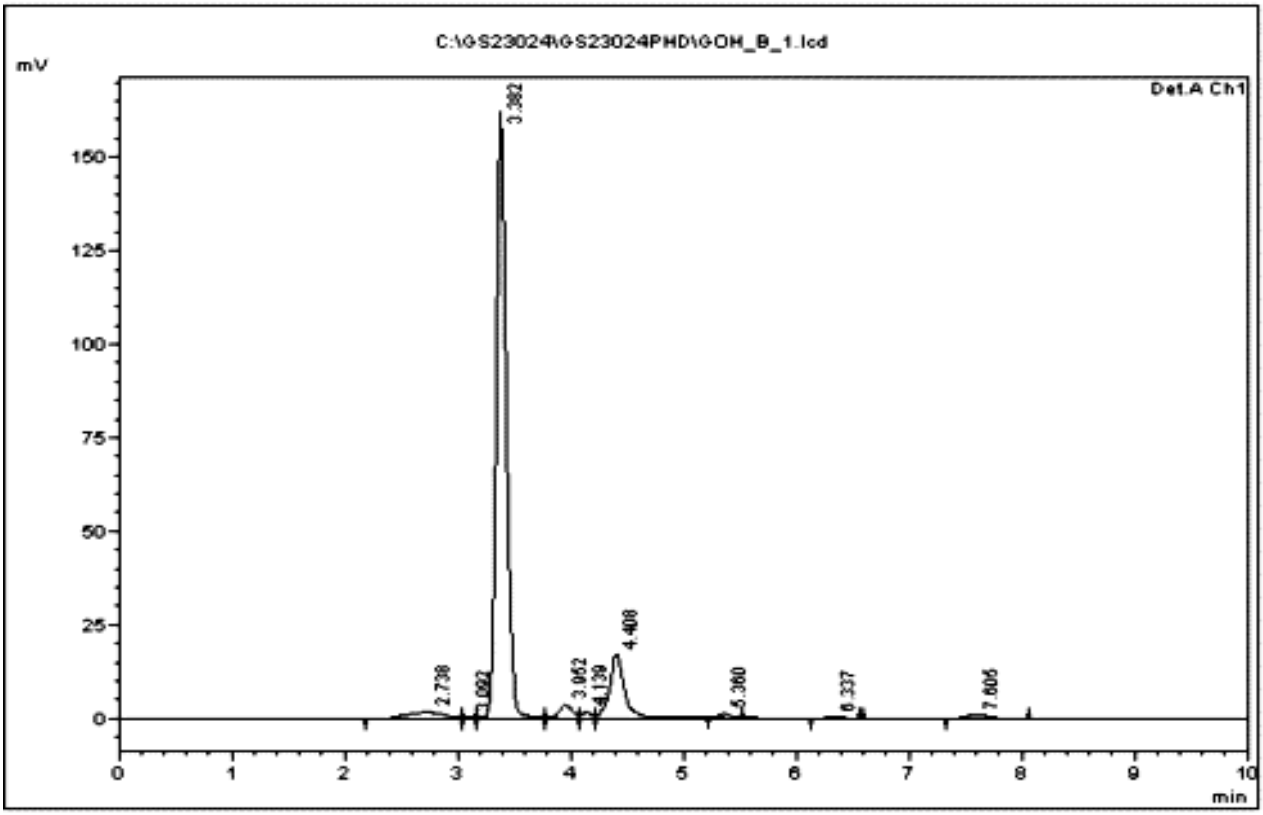
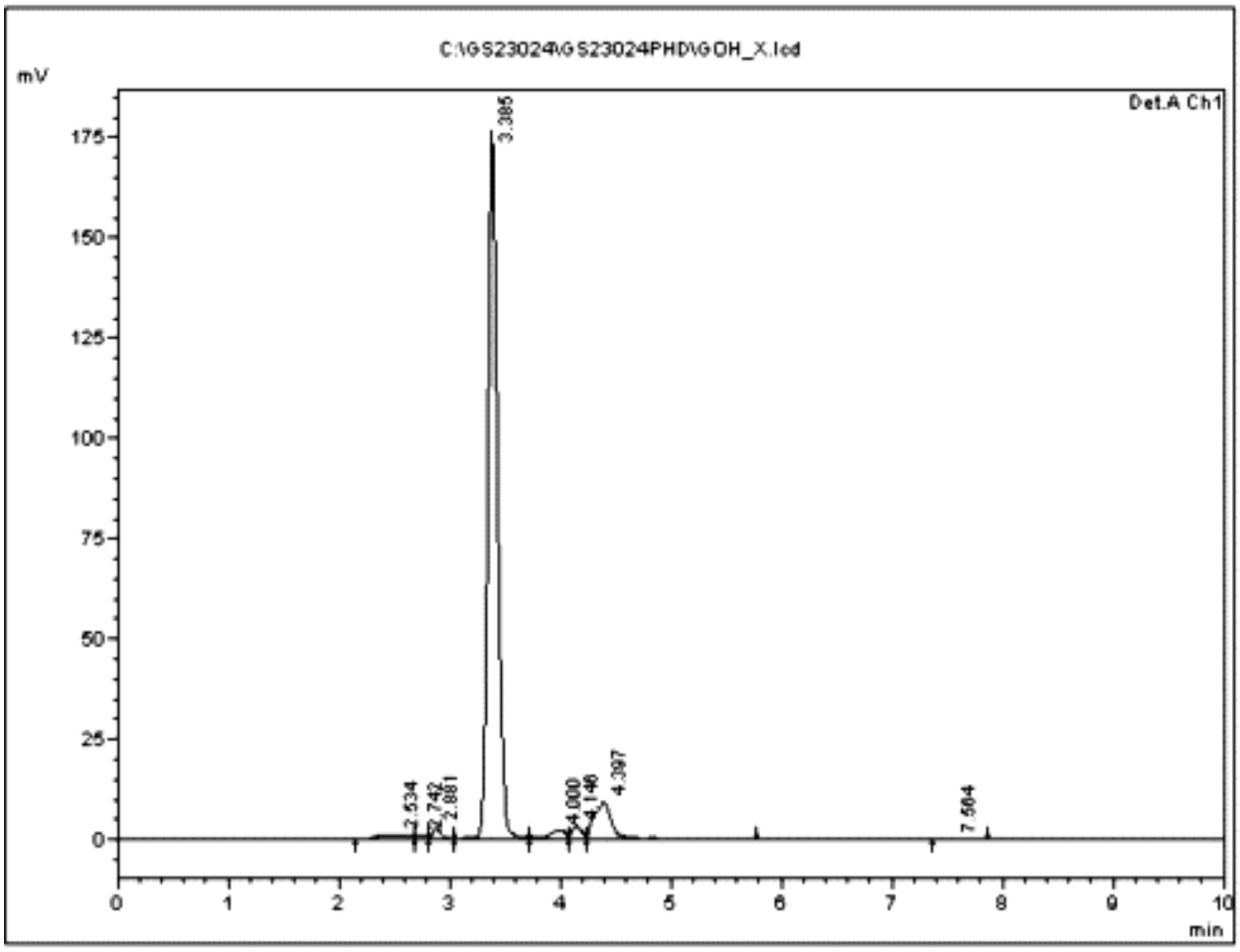


Peak Table

Detector A Ch1 254nm

PeakN	Ret. Time	Area	Height	Area %	Height %
1	2.738	39092	1846	3.051	0.977
2	3.092	2383	350	0.186	0.185
3	3.382	1013120	162096	79.059	85.794
4	3.952	30925	3561	2.413	1.885
5	4.139	10826	1663	0.845	0.880
6	4.408	162262	16967	12.662	8.980
7	5.360	8583	1205	0.670	0.638
8	6.337	2913	324	0.227	0.171
9	7.605	11375	926	0.888	0.490
Total		1281479	188937	100.000	100.000

Appendices: Figure A1. Example of HPLC chromatogram of methanol/water mixture with 1.1 mg BHET

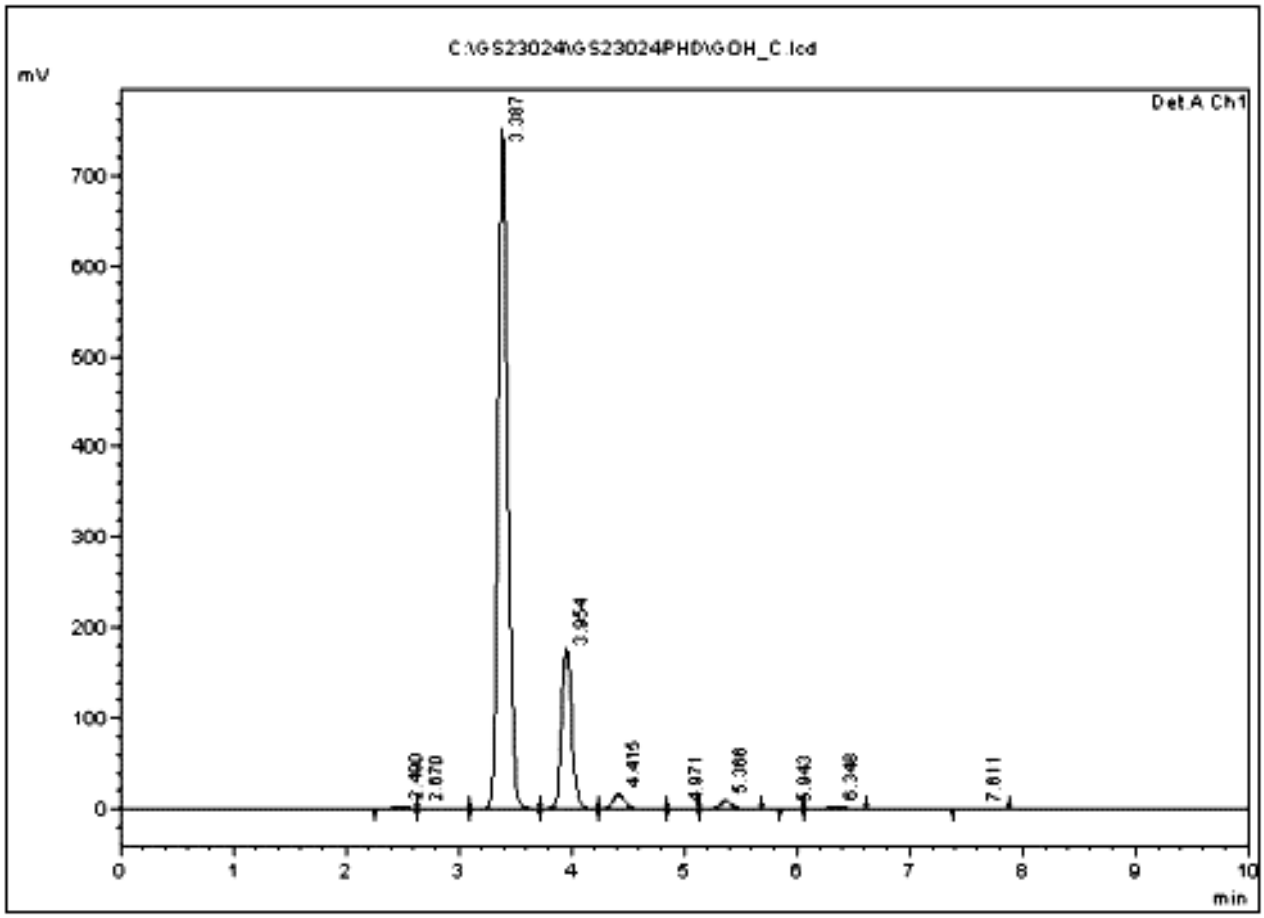


Peak Table

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	2.534	25979	1217	2.006	0.617
2	2.742	7025	981	0.543	0.497
3	2.881	16773	3142	1.295	1.592
4	3.385	1084934	176851	83.793	89.597
5	4.000	26826	2288	2.072	1.159
6	4.146	21985	3395	1.698	1.720
7	4.397	107815	9210	8.327	4.666
8	7.564	3447	302	0.266	0.153
Total		1294784	197386	100.000	100.000

Appendices: Figure A2. HPLC chromatogram of glycolysis product

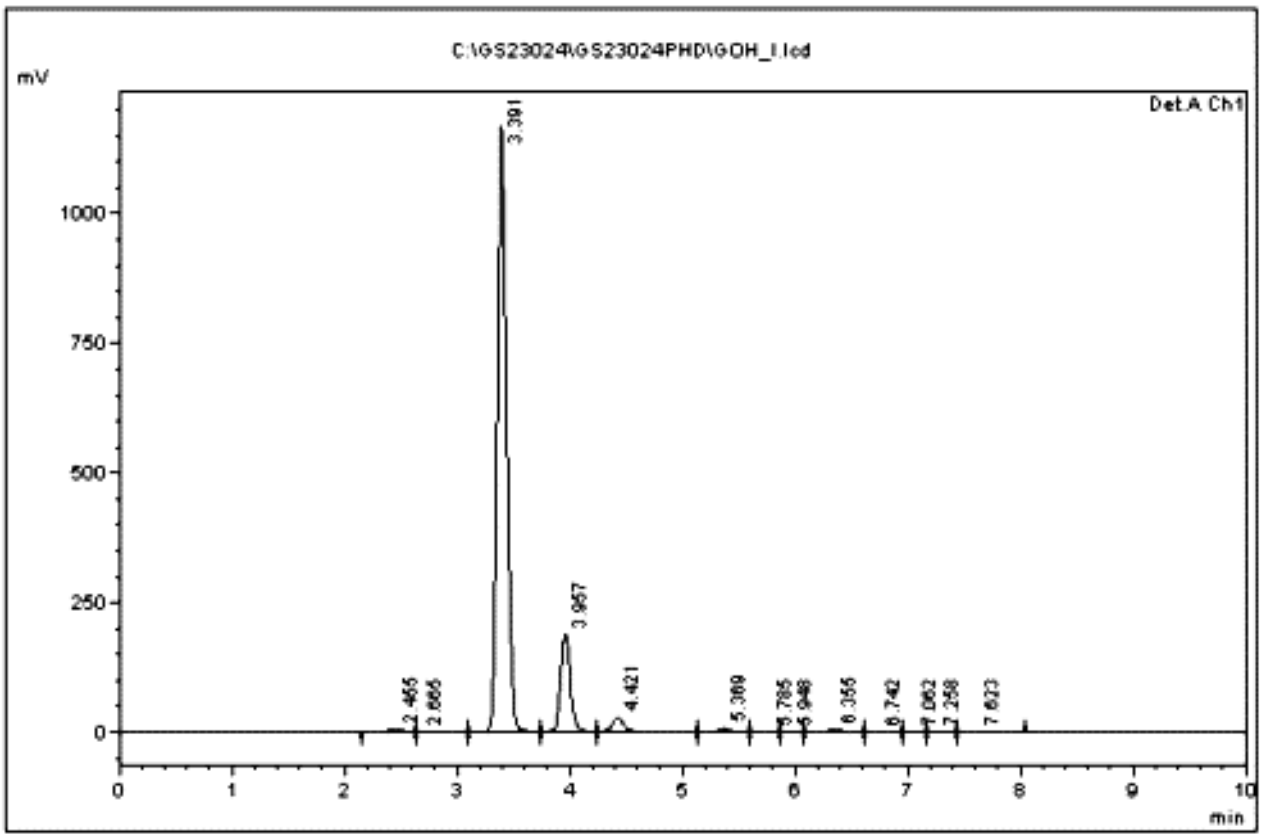


Peak Table

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	2.490	25829	2515	0.435	0.261
2	2.670	6940	781	0.117	0.081
3	3.387	4535614	752021	76.376	78.154
4	3.954	1125815	177133	18.958	18.409
5	4.415	130962	15942	2.205	1.657
6	4.971	3307	258	0.056	0.027
7	5.366	70664	9590	1.190	0.997
8	5.943	1416	140	0.024	0.015
9	6.348	34833	3576	0.587	0.372
10	7.611	3148	274	0.053	0.028
Total		5938528	962230	100.000	100.000

Appendices: Figure A3. HPLC chromatogram of CP obtained at 3 hours crystallization time

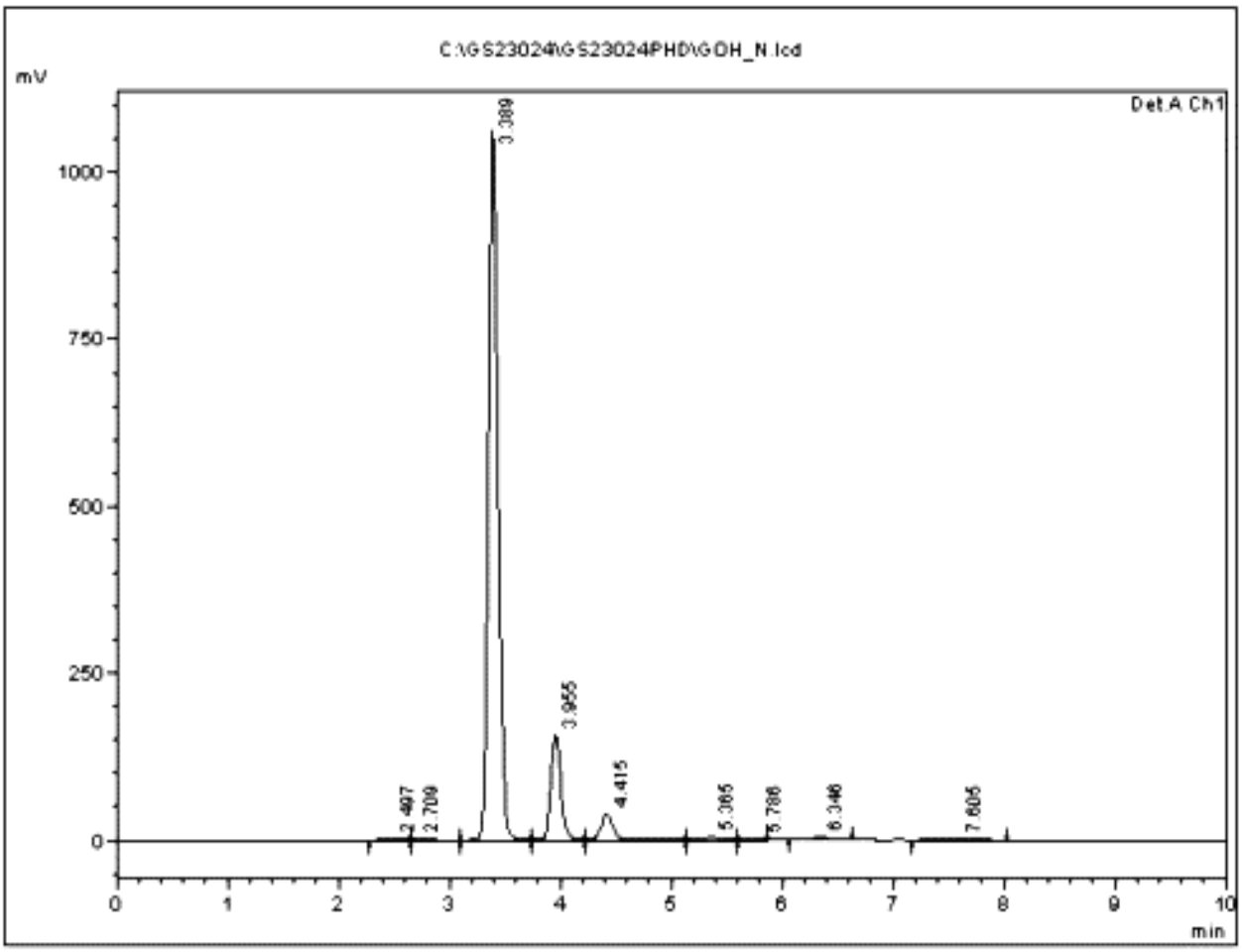


Peak Table

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	2.455	45587	4208	0.546	0.300
2	2.665	14380	813	0.172	0.058
3	3.391	6770895	1169988	81.138	83.295
4	3.957	1159733	189252	13.898	13.473
5	4.421	233159	27973	2.794	1.991
6	5.369	54197	6684	0.649	0.476
7	5.785	6085	396	0.073	0.028
8	5.948	4491	397	0.054	0.028
9	6.355	43444	4057	0.521	0.289
10	6.742	3932	220	0.047	0.016
11	7.062	1840	153	0.022	0.011
12	7.258	2041	135	0.024	0.010
13	7.623	5085	357	0.061	0.025
Total		8344868	1404631	100.000	100.000

Appendices: Figure A4. HPLC chromatogram of CP obtained at 2 °C crystallization temperature



Peak Table

Detector A Ch1 254nm

PeakN	Ret. Time	Area	Height	Area %	Height %
1	2.497	25474	2531	0.324	0.199
2	2.709	5420	494	0.069	0.039
3	3.389	6441681	1061385	81.876	83.562
4	3.955	994586	156163	12.642	12.294
5	4.415	301651	38389	3.834	3.022
6	5.365	37507	5066	0.477	0.399
7	5.786	1388	119	0.018	0.009
8	6.346	48325	5121	0.614	0.403
9	7.605	11578	917	0.147	0.072
Total		7867609	1270184	100.000	100.000

Appendices: Figure A5. HPLC chromatogram of CP obtained using mass ratio 5:1 of distilled water to GS