

## CHAPTER III EXPERIMENTAL

### 3.1 Chemicals

- Acetone, analytical grade, CARLO ERBA, Italy
- Glycerol, 99.5% W/V, VWR Prolabo, France
- Diglycerol, Solvay chemical, Germany
- Solketal, ACROS Organics, USA
- Sodium carbonate, CARLO ERBA, Italy
- Sodium hydroxide, RCI Lab-scan, Thailand
- Concentrated sulfuric acid (98%vol), RCI Lab-scan, Thailand
- Ethanol-water (95:5), HPLC grade, RCI Lab-scan, Thailand
- *p*-toluenesulfonic acid, CARLO ERBA, Italy
- Molecular sieve 3A, Fluka Analytical, Italy
- Nitrogen gas, Thailand
- Isopropanol, RCI Lab-scan, Thailand
- Benzyl alcohol, ACROS Organics, USA

### 3.2 Characterization Instruments

- Gas Chromatograph (GC), GC Agilent 6890
- Fourier Transform Infrared Spectroscopy (FT-IR), Thermo NICOLET NEXUS 670
- GC×GC Time-of-flight MS, LECO pegasus 4D, GC Agilent 7890

### 3.3 Methodology

#### 3.3.1 Preparation of Protected Glycerol (Solketal)

A mixture of glycerol, acetone, *p*-toluenesulfonic acid was a catalyst and molecular sieve 3A within desired ratio was reacted in glass reactor with total

reflux ratio at 75°C for 12 hours. After 12 hours of the reaction, the ice bath was used to cool down the temperature and then sodium carbonate was added for neutralization. And then, vacuum distillation was used to purify the product. The samples were taken at 2, 4, 6, 8, 10, 12 hour to analyze for the protected glycerol conversion by GC technique.

### 3.3.2 Preparation Standard Dibenzyl Ether

Benzyl alcohol was reacted with 2.2% by weight of *p*-toluenesulfonic acid. The reaction system was glass batch reactor with Dean and Stark apparatus at 110 °C for 14 hours. After reaction, the product was neutralized by sodium carbonate. After that, the samples were taken at 1, 2, 3 and 4 hour to estimate and analyze dibenzyl ether. The mixture product was analyzed by GC and GC×GC Time-of-flight MS technique.

### 3.3.3 Etherification between Solketal and Benzyl Alcohol

Solketal and benzyl alcohol were reacted with 2.2% by weight of *p*-toluenesulfonic acid in glass reactor with total reflux at 110 °C for 12 hours. After 12 hours of the reaction, the ice bath was used to cool down a temperature's product and then added sodium carbonate for neutralization the product. The samples were taken at 2, 4, 6, 8, 10, 12 hour to estimate and analyze the benzyl alcohol conversion and selectivity of benzyl solketal and by-products by GC and GC×GC TOF MS technique.

### 3.3.4 Product Analysis

For qualitative analysis, the products were identified by using FT-IR Thermo Nicolet Nexus 670. This FT-IR spectroscopy was used for test the chemical bond properties and structure preperities such as stretching and bending in the chemical bonds.

For quantitative analysis, the products were analyzed by using Gas chromatograph (GC), GC agilent 6890 with FID detector. Chromatogracpic column is Stabliwax column and inlet temperature is 250 °C. temperature program start at 40 °C and hold for 5 minutes. The ramp temperature is 5 °C per minute. The final temperature is 230 °C and hold for 17 minutes. Moreover, For GC×GC Time-of-flight MS is operated under the conditions in Table 3.1 and Table 3.2

**Table 3.1** GC×GC Time-of-flight MS condition

GC condition		MS condition	
1 <sup>st</sup> column	Rti-5sil MS	Start mass	30 $\mu$
2 <sup>nd</sup> column	DB-WAX	End mass	600 $\mu$
Carrier gas	Helium	Acquisition rate	200 spectra/s
FID	250 °C	Detector voltage	1300
Modulator temperature	15 °C	Electron energy	-70 volts
Chiller temperature	-80 °C	Ion source temperature	250 °C

**Table 3.2** Chromatographic temperature program for GC×GC Time-of-flight MS

Oven No.	Step	Rate (°C/min)	Target temperature (°C)	Duration (min)
1	1	Initial	40.00	5.00
and 2	2	10.00	230.00	7.00