



CHAPTER II

EXPERIMENTS

2.1 Instruments and equipment

Melting points were determined with Fisher-Johns melting point apparatus or electrothermal digital melting point apparatus, model IA 9100. Thin layer chromatography (TLC) was performed on aluminum sheets precoated with silica gel (Merck Kieselgel 60 PF254). The FT-IR spectra were recorded on Nicolet Fourier Transform Infrared Spectrophotometer, model Impact 410. Solid samples were incorporated to potassium bromide to form a pellet. The $^1\text{H-NMR}$ was performed in deuterated chloroform (CDCl_3) or acetone- d_6 or deuterated dimethylsulfoxide (DMSO-d_6) with tetramethylsilane (TMS) as an internal reference on a Bruker model ACF 200 spectrometer which was operated at 400 MHz for ^1H and 50.32 MHz for ^{13}C nuclei. The chemical shifts were assigned by comparison with residue solvent protons. $\text{CDCl}_3/\text{DMSO-d}_6$ means that DMSO-d_6 is added dropwise to a suspension of the compound in CDCl_3 until a clear solution is obtained. The surface tension and contact angle were recorded on a contact angle meter model Kruss DSA 10.

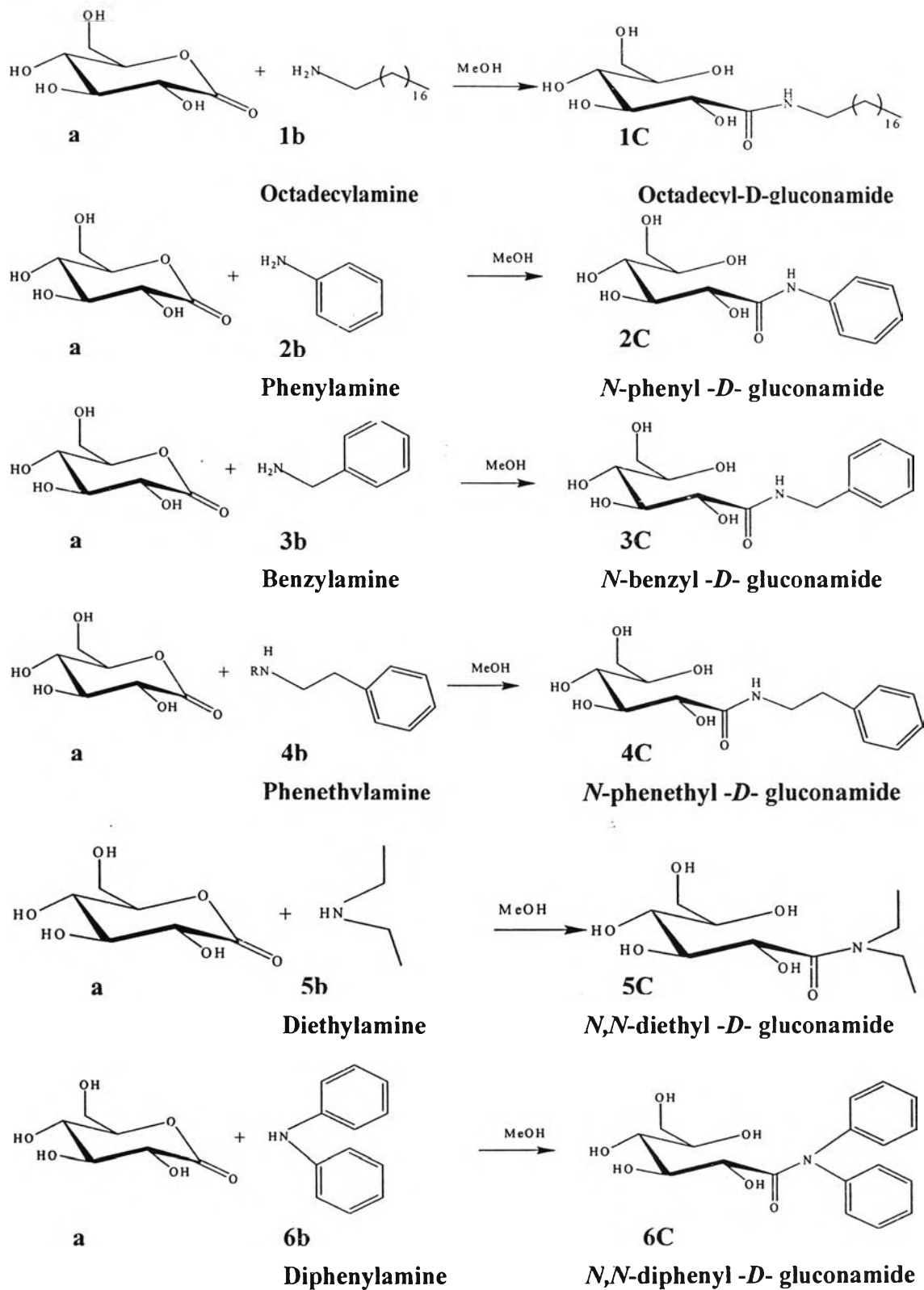
2.2 Chemicals

All solvents used in this research were purified prior to use by standard methodology except for those which were reagent grades. The reagents used for synthesizing the precursors, sugar esters and other compounds were purchased from Fluka and Cognis chemical companies or otherwise stated and were used without further purification.

2.3 Dipping reagent

Potassium permanganate in water were used for detecting spots of synthesized sugar compounds.

2.4 Synthesis

2.4.1 General synthesis of *N*-alkyl/aryl -*D*-gluconamides

In a 100 mL round bottle with stirrer, D-glucono-1,5-lactone (5.74 mmol) and a primary (**1b**, **2b**, **3b** and **4b**) or secondary amine (**5b** and **6b**) (6.65 mmol) were dissolved in methanol (20 mL) at room temperature. The reaction mixture was stirred for 24 hr and then the reaction was performed with reflux. After the reaction was completed, white precipitate (**1C**, **2C**, **3C** and **4C**) was obtained. The precipitates were removed by filtration and washed with acetone.

For **5C** and **6C** after the reaction was completed, yellow and violet solutions were obtained, respectively. By product and excess MeOH were evaporated to gain the reaction crude as yellowish oil and violet powder, respectively.

All products were ground and dried in a vacuum desiccator. The yields of the precipitated products varied from 40 – 86 % [45,46 and 53].

2.4.2 ¹H NMR and IR spectral data of *N*-alkyl/aryl -D-gluconamides (**1C-6C**)

The following spectral data are all common for D-gluconamide **1C-6C**; ¹H NMR spectra were performed in deuterated chloroform or dimethylsulfoxide-d₆ or acetone- d₆ with tetramethylsilane as an internal reference on Bruker model ACF200 spectrometer and Varian model Mercury 400 spectrometer. The solvent was the reference in all measurement. Only the protons that are significant to the derivatives were reported. Infrared (IR) spectra were recorded on a Nicolet Fourier Transform Infrared spectrometer, and all sample were prepared with KBr. Only the characteristic spectral peaks of new compounds are presented. The percent yield was calculated and presented in **Appendix B.1**

2.4.3 Determination gluconamide derivatives (**1C-6C**) by TLC

After developing and drying TLC plates were spotted substrates (D-glucono-1,5-lactone) and compounds (**1C – 6C**) in 1 : 1 ethyl acetate : hexane solvent system. TLC plates were dried and dipped in Potassium permanganate solution and dried again, the spots were appeared and compare the Retention Factor (R_f) values of compounds run on the same plate at the same time.

2.5 Characterization

The properties of all studied compounds were compared with two reference surfactants: nonylphenol ethoxylates of 4 and 40 ethylene oxide units (Cognis, Germany). Stock solutions of 1% w/v of the surfactant and references in distilled water were

prepared and used within the individual measurements unless other concentrations are stated.

2.5.1 Surface tension measurement

Solutions of the synthesized surfactants in water with concentrations of 0.1 and 1.0% w/v were prepared. The surface tension of surfactant solution was determined by using contact angle meter model Kruss DSA 10, Germany.

2.5.2 Solubility

Solutions of the surfactants synthesized in water with concentration of 1% w/v were prepared. The surfactants (0.1 g) were diluted in water up to 10 mL. If all surfactants were clearly dissolved, the solubility limit was determined to be over 1% w/v. In cases where the solubility limit was below concentrations of 1% w/v, the outcome was studied gravimetrically for aqueous solutions.

Solubility in organic solvents (ethanol and dodecane) were studied at three different concentrations: 0.01, 0.15 and 1 % w/v with the result indicating in which concentration of the surfactant that was fully soluble. From these tests the solubility range was determined for each surfactant. The solubility of these samples was studied by visual opacity [54].

2.5.3 Wetting

Wettability can be determined by measuring contact angle where the surface of a liquid droplet and the solid surface are in contact. A liquid with high surface tension will form a droplet on a surface, where a liquid with low surface tension will spread out a surface.

The wetting properties of the surfactants were determined by comparison with NP4 and NP40 and with pure water. The contact angles of references were measured by with four or five repetitions using contact angle meter (model KRUSS DSA10, Germany). A wetting property scale from 0 to 6 was used 98° (for 0), 66° (for 1), 55° (for 2), 43° (for 3), 31° (for 4), 28° (for 5) and 0° (for 6), which the properties of the references are defined as the values 5, 4 and 0 for NP-4, NP-40 and pure water, respectively. The wetting properties of surfactants were compared with the references and given values ranging from 0 to 6 [55].

With a micropipette, a droplet (30 μL) of the stock solution (1 wt/vol% or the maximal saturation concentration, whichever is lower) was put onto a parafilm and the angle was calculated by the KRUSS software and reported the contact angle.

2.5.4 Foaming (ross- miles method : ASTM D 1173 – 63)

Aqueous solutions of the surfactants and reference surfactants (40 mL 0.1 wt/vol% surfactant) were prepared. The solution was poured into a 100-mL graduated cylinder. The cylinder containing the solution was turned upside down a total of 10 times at a rate of 1 turn 2 s. The foam produced (in mm) was measured immediately and after 1 min. These data were used for estimating the foamability and foam stability of the surfactants. All experiments were performed at least five times, and the average of the measurements was used for evaluations [16].