RAMIZAH BINTI RAMLI

MASTER OF SCIENCE

2017

SYNTHESIS AND CHARACTERIZATION OF SUBSTITUTED-BENZOYLTHIOUREA VALINE AND THREONINE DERIVATIVES

RAMIZAH BINTI RAMLI

MASTER OF SCIENCE
UNIVERSITI MALAYSIA TERENGGANU

2017

SYNTHESIS AND CHARACTERIZATION OF SUBSTITUTED-BENZOYLTHIOUREA VALINE AND THREONINE DERIVATIVES

RAMIZAH BINTI RAMLI

Thesis Submitted in Fulfillment of the Requirement for Degree of Master of Science in the School of Fundamental Sciences Universiti Malaysia Terengganu

April 2017

Abstract of thesis presented to the Senate of Universiti Malaysia Terengganu in fulfillment of the requirement for the degree of Master of Science.

SYNTHESIS AND CHARACTERIZATION OF SUBSTITUTED-BENZOYLTHIOUREA VALINE AND THREONINE DERIVATIVES

RAMIZAH BINTI RAMLI

April 2017

Main Supervisor : **Associate Professor Mohd Sukeri Mohd Yusof, Ph.D.**

Co- Supervisors : **Siti Kamilah Che Soh, Ph.D.**
Associate Professor Nurziana Ngah, Ph.D.

School : **School of Fundamental Sciences**

Study of benzoylthiourea derivatives has received immense attentions from researchers worldwide due to their versatility in many fields. Owing to the great potentials they offered, therefore, this research project was carried out. In this study, twenty substituted-benzoylthiourea amino acid have been successfully synthesized and characterized using FT-IR spectroscopy, UV-Vis spectrophotometer, NMR spectroscopy and Mass Spectrometry. To be specific, sixteen new compounds have been obtained, while four compounds have been previously reported. Compound 4-MeOBTT was obtained as single crystal and it has been further characterized by Single Crystal X-Ray Crystallography. The FT-IR spectra demonstrated the presence of six important absorption bands of *ν*(N-H), *ν*(O-H), *ν*(C=O-OH), *ν*(C=O-NH), *ν*(C=C) and *ν*(C=S) observed within range of 3171-3486 cm^{-1}, 2962-3302 cm^{-1}, 1703-1768 cm^{-1}, 1655-1703 cm^{-1}, 1499-1560 cm^{-1} and 703-795 cm^{-1}, respectively. Meanwhile, UV-Vis spectra exhibited the presence of two chromophores corresponded to the n-π* and π-π* transitions. Proton NMR spectra displayed all of the expected resonances of (-C=O-N*H*) and (-C=S-N*H*) appeared in the range of δ_H 9-12 ppm and 10-11 ppm. Whereas, the ^{13}C NMR spectra showed the existence of three main resonances of C=S, C=O-OH and C=O-NH appeared downfield at

approximately δ_C 180-182 ppm, 170-175 ppm and 165-171 ppm, respectively. Mass spectra obtained proved the presence of molecular ion peaks at the expected mass to charge ratio. From the X-ray crystallography analysis, only one molecular structure has been obtained which was for compound 4-MeOBTT. Two independent molecules in an asymmetric unit have been stabilized by intramolecular hydrogen bonds. The packing diagram showed the presence of intermolecular hydrogen bonds viewed down the *a* axis. All of the bond lengths and angles were in normal ranges. As for biological application, all of the compounds were screened for their antibacterial activity against *Staphylococcus aureus*, *Staphylococcus epidermidis*, *Salmonella typhimurium* and *Escherichia coli*. Result obtained revealed that no compounds were able to inhibit the growth of the tested bacterial strains.

Abstrak tesis yang dikemukakan kepada Senat Universiti Malaysia Terengganu sebagai memenuhi keperluan untuk ijazah Sarjana Sains.

SINTESIS DAN PENCIRIAN SEBATIAN BENZOILTIOUREA PENUKARGANTI VALINA DAN TREONINA

RAMIZAH BINTI RAMLI

April 2017

Penyelia Utama : Profesor Madya Mohd Sukeri Mohd Yusof, Ph.D.

Penyelia Bersama : Siti Kamilah Che Soh, Ph.D.
Profesor Madya Nurziana Ngah, Ph.D.

Pusat Pengajian : Pusat Pengajian Sains Asas

Kajian terhadap terbitan benzoiltiourea telah mendapat perhatian begitu ramai pengkaji dari seluruh dunia disebabkan oleh kecenderungannya dalam pelbagai bidang. Berlandaskan potensi yang ada pada terbitan benzoiltiourea, kajian ini telah dijalankan. Dalam kajian ini, dua puluh penukarganti asid amino benzoiltiourea telah berjaya disintesis dan dicirikan menggunakan spektroskopi Infra-Merah, spektrofotometer Ultralembayung-Nampak, spektroskopi Resonan Magnetik Nuklear (RMN) dan Spektrometri Jisim. Secara khususnya, enam belas sebatian baru telah diperoleh, manakala, empat sebatian lagi telah dilaporkan sebelum ini. Sebatian 4-MeOBTT telah diperoleh dalam bentuk hablur tunggal dan telah dicirikan lagi dengan Kristallografi Sinar-X Hablur Tunggal. Spektra infra-merah menunjukkan kehadiran enam serapan penting iaitu *v*(N-H), *v*(O-H), *v*(C=O-OH), *v*(C=O-NH), *v*(C=C) dan *v*(C=S), masing-masing pada 3171-3486 cm^{-1}, 2962-3302 cm^{-1}, 1703-1768 cm^{-1}, 1655-1703 cm^{-1}, 1499-1560 cm^{-1} dan 703-795 cm^{-1}. Spektra ultralembayung-nampak menunjukkan kehadiran dua kromofor yang disebabkan oleh peralihan elektronik n-π^* dan π-π^*. Spektra ^{1}H RMN mengesahkan kehadiran kesemua signal proton (-C=O-N*H*) and (-C=S-N*H*) pada julat δ_H 9-12 ppm dan 10-11 ppm. Spektra ^{13}C RMN pula menunjukkan kewujudan tiga puncak utama iaitu

C=S, C=O=OH dan C=O-NH pada δ_C 180-182 ppm, 170-175 ppm dan 165-171 ppm. Spektra jisim bagi semua sebatian membuktikan kehadiran puncak ion molekular pada nisbah jisim kepada cas yang diharapkan. Menerusi analisis kristalografi sinar-X, hanya satu struktur molekul yang berjaya diperoleh iaitu bagi sebatian 4-MeOBTT. Unit asimetrik yang mengandungi dua molekul bebas telah distabilkan oleh ikatan hidrogen intramolekul. Gambarajah padatan menunjukkan kehadiran ikatan hidrogen intermolekular apabila dilihat dari bawah paksi *a*. Kesemua panjang ikatan dan sudut ikatan berada dalam julat yang normal. Bagi aplikasi biologi pula, kesemua dua puluh sebatian telah disaring bagi melihat aktiviti antibakteria terhadap *Staphylococcus aureus*, *Staphylococcus epidermidis*, *Salmonella typhimurium* dan *Escherichia coli*. Keputusan yang diperoleh mendapati tiada sebarang sebatian yang mampu merencatkan pertumbuhan bakteria yang diuji.