UNIVERSITI TEKNOLOGI MARA

COUMARINS - FAST SYNTHESIS BY THE KNOEVENAGEL CONDENSATION UNDER MICROWAVE IRRADIATION

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Dissertation submitted in partial fulfillment of the requirement for the degree of Bachelor of Pharmacy (Hons)

Faculty of Pharmacy

November 2008

ACKNOWLEDGEMENT

I couldn't be more grateful now that I have finished my thesis writing. First and foremost I would like to thank Allah for his blessings and the strength he's given me to finish this research.

I would like to specially thank my research supervisor, Dr Humera Naz for her guidance and kindness in making this research a success. Also, to Dr Fareeda Feroz, Dr Sadia Sultan and Dr Syed Adnan Ali Shah, thank you very much.

I owe my thanks also to all members of Institut Kajian Ubat Semulajadi (iKUS) especially Mr Mohd Faizal Mohd Nasir for helping me out with the machines and during experiments. To all my colleagues, I must say you make this all possible.

Last but not least, I will not forget my family and friends with whom I find my courage and strength to complete this job. I am blessed by their unconditional love which has indeed given me motivation to stay focused and strong in completing this research. Thank you very much.

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ABSTRACT

Coumarin nowadays is of great importance especially in the area of pharmaceutical. Therefore, a faster way of producing coumarin was needed while maintaining the purity and yields of product as high as possible. One of the methods is Knoevenagel condensation under microwave assisted irradiation. This research was aimed to prove that under the microwave irradiation the Knoevenagel condensation can be successfully applied to the synthesis of a number of coumarins. Also, the researcher wanted to find the most suitable base for this reaction. 2-hydroxy-4-methoxy benzaldehyde and 2-hydroxy-5-methoxy benzaldehyde were reacted with diethyl melonate in the presence of three different bases; diethylamine, pyridine, and potassium *tert*-butoxide for three different reactions. The reactions were set under different conditions: stirring with no heating for six hours; stirring with heating at 80 °C for six hours; and under microwave assisted irradiation at 1000 watt power, 70 ^{0}C, and stirring speed of 100 rpm for one hour. TLC was used to prove the reactions were completed. To confirm the structure of coumarins (**13** and **16**) formed, ^{1}H and ^{13}C-NMR analyses were used. From the results, it was found out that diethylamine was the most suitable base for this reaction therefore used throughout the research. Productions of coumarins were significantly increased from stirring, no heating condition (29.07 % and 61.13 %) to stirring, heating condition (39.52 % and 68.23 %) to microwave assisted irradiation (48.39 % and 84.68 %). The time for reaction under microwave condition was also decreased to one hour and yet the percentage yield was still maintained.

CHAPTER 1

INTRODUCTION

1.1 Introduction to Coumarins

Coumarins include a very large class of phenolic substances which can be found in plants and consists of an aromatic ring fused with pyrone rings (condensed lactone ring) (Hoult and Paya, 1996). At least 1300 derivatives of coumarins have been identified until now, principally as secondary metabolites in green plants but also in fungi and bacteria (Murray *et al.*, 1982). Coumarins are widely distributed in plants, and are commonly found in families such as the Umbellliferae/Apiaceae and Rutaceae, both in the free form and as glycosides. Coumarin itself is found in sweet clover (Melilotus species; Leguminosae/Fabaceae) (Dewick, 2003). Coumarin gives a pleasant smell and gives a characteristic odour to hay. Because of their characteristic smells, other simple coumarins are sometimes exploited in perfumery, but these are lost if the molecules are conjugated to sugars or acids-a frequent occurrence in nature. Substitution on coumarins can occur at many sites. There are many possible permutations offered by substitution and conjugation, and this readily explains why so many of these substances occur naturally (Hoult and Paya, 1996). Coumarin has been found to be freely soluble in ethanol, chloroform, diethyl ether and is slightly soluble in water (Cohen, 1979).