THE DETERMINATION OF CAFFEINE CONTENT IN TEA USING HEADSPACE SOLID PHASE MICROEXTRACTION (HS-SPME) AND GAS CHROMATOGRAPHY-MASS SPECTROMETRY (GC-MS)

NUR AMIRAH BINTI MOKHTAR

Final Year Project Report Submitted in Partial Fulfillment of the Requirement for the Degree of Bachelor of Science (Hons) Chemistry (Forensic Analysis) in the Faculty of Applied Sciences Universiti Teknologi MARA

JULY 2015

ACKNOWLEDGEMENTS

In the name of Allah, The Most Gracious and The Most Merciful. Peace and blessing of Allah The Al-Mighty to our beloved, final Prophet Muhammad s.a.w and his relatives, all his companions and those who have followed. Alhamdulillah, all praise and thankfulness to Allah s.w.t, for His blessings and for giving me strength and opportunity in completing this project within its expectation and time allocated.

First of all, I would like to express my deepest thanks and appreciation to my supervisor, Assoc. Prof. Zuraidah Abdullah Munir for her understanding, infinite guidance, valuable suggestion and continuous support throughout the planning and execution of the project. Without her knowledge, generosity and great amount of patience, this project would not be a success.

Special thanks go to the lab assistant Mr. Ahmad Kambali Khalil, who is willing to teach and assist me on how to handle the GC and for his help in every aspect of this project. The knowledge and advice given, I will remember till the end of the days.

I would also like to extend my gratitude to my beloved family for their understanding, encouragement, financial support and motivation throughout my study. Not forgetting, my deepest thanks to all my classmates and friends for their helpfulness while completing this project. Thanks a millions. I hope this project will be beneficial and useful to all.

Nur Amirah binti Mokhtar

TABLE OF CONTENTS

Page

ACKN TABLI LIST C LIST C ABSTH ABSTH	OWLED E OF CO OF TABI OF FIGU OF ABBI RACT RAK	GEMENTS INTENTS LES RES REVIATION	۹S	iii iv vi vii viii ix x
CHAP	FER 1 II	NTRODUCT	ΓΙΟΝ	
1.1	Backgı	ound of stud	У	1
1.2	Proble	n statements		3
1.3	Signifi	cance of stud	l y	4
1.4	Object	ives of study	· · · · · · · · · · · · · · · · · · ·	5
CHAP	FER 2 L	ITERATUR	E REVIEW	
2.1	Histori	cal backgrou	nd of tea	6
2.2	Types of tea			
	2.2.1	Black tea		9
	2.2.2	Oolong te	a	10
	2.2.3	Green tea		10
2.3	Caffeir	ne		11
2.4	Previous studies of caffeine in tea			13
	2.4.1	Extraction	techniques of caffeine in tea	13
		2.4.1.1	Ultrasound assisted extraction-dispersive liquid-	14
			liquid microextraction (UAE-DLLME)	
		2.4.1.2	High pressure processing (HPP)	15
		2.4.1.3	Solid phase microextraction (SPME)	16
	2.4.2	Analysis o	of caffeine in tea	17
		2.4.2.1	High performance liquid chromatography (HPLC)	18
		2.4.2.2	Gas chromatography-mass spectrometry (GC-MS)	19

CHAPTER 3 METHODOLOGY

3.1	Materials and chemicals		
3.2	Instrument		
3.3	Standard preparation		
3.4	Sample preparation		
	3.4.1 Solid phase microextraction (SPME) procedure	22	
3.5	Quantitative analysis by gas chromatography-mass spectrometry (GC-		
	MS)	23	

ABSTRACT

THE DETERMINATION OF CAFFEINE CONTENT IN TEA USING HEADSPACE SOLID PHASE MICROEXTRACTION (HS-SPME) AND GAS CHROMATOGRAPHY-MASS SPECTROMETRY (GC-MS)

This study was conducted to determine the caffeine content in different types and brands of tea which are black tea, oolong tea and green tea and to identify the major volatile compounds present in those tea by using headspace-solid phase microextraction (HS-SPME) with gas chromatography-mass spectrometry (GC-MS). About 1 g of dry tea leaves from tea samples were extracted by using polydimethylsiloxane/divinylbenzene (PDMS/DVB) fiber at 50 °C for 40 min. The quantification of caffeine was calculated based on calibration curve of standard caffeine. The result shows that green tea has the highest amount of caffeine, followed by oolong tea and black tea. While in the analysis of four brands of black tea, brand D has the highest caffeine content, followed by brand C, brand B and brand A. The major volatile compounds present in black tea, oolong tea and green tea are benzaldehyde, D-limonene, cyclopentasiloxane, methyl salicylate, tetradecane and caryophyllene. In this study, D-limonene is only present in green tea. Therefore, it can be used as the reference volatile compound to differentiate between unfermented green tea and fermented tea such as black tea and oolong tea.

CHAPTER 1

INTRODUCTION

1.1 Background of study

Tea is known as the second most popular beverages in the world after water. Tea has a variety of types and all types of tea are made from the leaves of plant species of *Camellia sinensis* and it belongs to the *Theaceae* family (Sereshti *et al.*, 2013). Generally, teas are classified into three categories which are fully-fermented black teas, semi-fermented oolong teas and unfermented green teas (Wang *et al.*, 2008). Black tea is the most popular type of tea consumed by people around the world.

Malaysia has its own tea plantation and Cameron Highlands is one of the biggest tea plantations in Asia. The biggest production of tea in Cameron Highlands is black tea, followed by oolong tea and green tea. The famous brands of black tea in Malaysia include Boh, Lipton and Sabah Tea. The fermentation or oxidation process of black tea causes the dark brown colour to the tea. Meanwhile the green tea retains the green colour of the leaves due to its unfermented process. Oolong tea is known as semi-fermented tea and the colour is quite similar to the black tea.

1