EFFECT OF DIFFERENT SPECIES, PROCESSING PROCEDURE AND DEGREE OF ROASTING ON VOLATILE COMPOUNDS PRODUCTION IN THAI COFFEE

By

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การศึกษาผลของสายพันธุ์ (อาราบิก้า และโรบัสต้า) กระบวนการผลิต และระดับการคั่ว (ระดับอ่อน กลาง และเข้ม)ที่แตกต่างกัน ต่อการเกิดสารระเหยในกาแฟไทย ผลของกระบวนการแปรรูปเมล็ดกาแฟ 4 วิธี ได้แก่ การแปรรูปแบบแห้งด้วยแสงอาทิตย์ การแปรรูปแบบแห้งด้วยเครื่องอบแห้ง การแปรรูปแบบเปียกแล้วลดความชื้นเมล็ดด้วยแสงอาทิตย์ และการแปรรูปแบบเปียกแล้วลดความชื้นเมล็ดด้วยเครื่องอบแห้ง ต่อองค์ประกอบทางเคมีของกาแฟสายพันธุ์อาราบิก้า โดยการวิเคราะห์ปริมาณไขมัน สารประกอบไนโตรเจน น้ำตาล และ ความเป็นกรด ในตัวอย่างเมล็ดกาแฟดิบ และเมล็ดกาแฟคั่ว พบว่า เมล็ดกาแฟดิบที่ผ่านกระบวนการแปรรูปที่แตกต่างกันจะมีปริมาณไขมัน และความเป็นกรดที่แตกต่างกัน ($p<0.01$) แต่มีปริมาณของสารประกอบไนโตรเจน และน้ำตาลไม่แตกต่างกัน ($p>0.01$) เมื่อศึกษาผลของระดับการคั่ว 3 ระดับ ได้แก่ ระดับอ่อน กลาง และเข้ม ต่อองค์ประกอบทางเคมีของเมล็ดกาแฟดิบสายพันธุ์อาราบิก้าที่ผ่านกระบวนการแปรรูปแบบเปียกแล้วลดความชื้นเมล็ดด้วยแสงอาทิตย์ พบว่า เมล็ดกาแฟที่ผ่านกระบวนการคั่วในระดับที่แตกต่างกันจะมีองค์ประกอบทางเคมีที่แตกต่างกัน ($p<0.01$)

การศึกษาองค์ประกอบของสารระเหยด้วยเทคนิค Purge & Trap / GC-MS ของกาแฟ 2 สายพันธุ์ ได้แก่ สายพันธุ์อาราบิก้า และโรบัสต้า ต่อองค์ประกอบของสารระเหยในเมล็ดกาแฟที่ผ่านการคั่วในระดับเข้ม พบว่า กาแฟสายพันธุ์อาราบิก้ามีปริมาณของสารในกลุ่ม furans และ pyridines สูง ส่วนโรบัสต้าจะประกอบด้วยสารระเหยในกลุ่มของalcohols pyrroles และ acids ได้แก่ butanoic acid เมล็ดกาแฟดิบที่ผ่านกระบวนการแปรรูปที่แตกต่างกัน พบว่า การลดความชื้นด้วยเครื่องอบแห้งสามารถกำจัด hexanal และ benzaldehyde จากเมล็ดกาแฟดิบ ซึ่งช่วยลดปริมาณองค์ประกอบของสารระเหยที่ไม่ต้องการได้ ตัวอย่างเมล็ดกาแฟที่ผ่านกระบวนการคั่วจะมีปริมาณของสารระเหยในกลุ่มของ furans pyrazines และ pyridines เพิ่มขึ้นเมื่อระดับการคั่วเพิ่มขึ้น เมล็ดกาแฟที่คั่วในระดับเข้มสารระเหยในกลุ่มของ pyrazine pyrroles และ acetic acid จะหายไป แต่จะพบ 2-methoxyphenol ซึ่งไม่พบในเมล็ดกาแฟที่คั่วระดับอื่น

การทดสอบโดย Linear Discrimination Analysis (LDA) พบว่ากราฟที่ได้สามารถนำไปใช้ในการทำนายและวิเคราะห์จัดกลุ่มตัวอย่างได้ถูกต้องในระดับ 90 ถึง 100 เปอร์เซ็นต์ เมื่อนำข้อมูลของสารระเหยจาก GC-MS มาเปรียบเทียบกันโดยใช้เทคนิค Partial Least Squares (PLS) Data Compression จะทำให้สามารถเห็นความแตกต่างของสารระเหยได้อย่างชัดเจนในเมล็ดกาแฟคั่วที่ต่างสายพันธุ์ อีกทั้งกระบวนการผลิตแบบแห้งและแบบเปียกที่ลดความชื้นด้วยแสงอาทิตย์ และระดับการคั่วที่แตกต่างกันก็มีผลต่อการเกิดขึ้นของสารระเหยในตัวอย่างเมล็ดกาแฟคั่วอย่างชัดเจน ตัวอย่างที่ผ่านกระบวนการแปรรูปแบบเดียวกันแต่ผ่านกระบวนการลดความชื้นที่แตกต่างกันจะให้องค์ประกอบของสารระเหยในตัวอย่างเมล็ดกาแฟคั่วไม่แตกต่างกัน ดังนั้นจึงสามารถใช้เครื่องอบแห้งเมล็ดกาแฟแทนการตากแดดในกระบวนการผลิตได้

ภาควิชาเทคโนโลยีอาหาร บัณฑิตวิทยาลัย มหาวิทยาลัยศิลปากร ปีการศึกษา 2547

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ลายมือชื่ออาจารย์ผู้ควบคุมวิทยานิพนธ์ 1. .... 2. .... 3. ... .

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The aim of this study was to investigate the effect of species (arabica and robusta), green coffee processing and degree of roasting (light, medium and dark) on volatile compounds production in Thai coffee. The green arabica coffee beans were processed by four different methods, dry process and drying with solar dry, dry process and drying with tray dryer, wet process and drying with solar dry and wet process and drying with tray dryer. Then, the chemical compositions, fat content, nitrogen compounds, total sugars and total acidity were determined in both green and roasted coffee beans. The results found that green arabica coffee beans prepared from different processing showed significant different of fat content and total acidity ($p<0.01$) but they were not significant different ($p>0.01$) of nitrogen compounds and total sugars. Green beans prepared by the same method (wet process and drying with solar dry) but different degrees of roasting (light, medium or dark) were determined the chemical compositions and it was found that degrees of roasting resulted in significant different ($p<0.01$) of chemical compositions.

Volatile compounds production was investigated by using purge and trap and GC-MS. The species affected the volatile compounds of coffee arabica and robusta. The highest amount of volatile compounds in dark roasted arabica was furans and pyridines, while robusta contained alcohols, pyrroles and butanoic acid. Only green beans prepared from dry and wet processes and drying method with tray dryer were eliminated hexanal and benzaldehyde which decreased undesirable volatile compounds. After roasting, furans, pyrazines and pyridines were increased as the increase of roasting degree from light to medium. At dark roasted degree, some volatile compounds, such as pyrazines, pyrroles and acetic acid were disappeared, but 2-methoxyphenol was found.

Predictive models were developed to classify the volatile compounds of coffee samples, using discriminant analysis. Linear discriminant analysis (LDA) was used to discriminate aroma coffee samples. The optimum classification of 90 to 100% was achieved in validation set, using linear discriminant analysis with the partial least squares (PLS) data compression technique. PLS plots shown significant differences of volatile compounds between arabica and robusta. Furthermore, the different degrees of roasting arabica from wet process with solar drying showed discriminately volatile compounds between roasted arabica at light, medium and dark roasted degree. The volatile compounds of medium roasted arabica from wet and dry process shown different plot locations. The discriminant analysis between solar drying and tray dryer method was not different of volatile compounds profiles. So, it was more advantage to use tray dryer instead of solar drying in coffee processing.

Department of Food Technology Graduate School, Silpakorn University Academic Year 2004

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CHAPTER 1

INTRODUCTION

Coffee beans are the seeds that occur in pairs in the red cherry-like fruits of an evergreen shrub belonging to the family *Rubiaceae* and the genus *Coffea* (Sivetz, 1963). *Coffea* growns wild in Africa and Madagascar and the genus includes a large number of species (Varnam and Sutherland, 1994). Only two species, *Coffea arabica* and *Coffea canephora* (robusta) have been successfully used in commercial cultivation (Clark, 1985). *Coffea arabica* cultivated in the northern part and coffea robusta in the southern part of Thailand (Jirasawat, 2003).

The factor affect coffee flavor such as the species, cultivation, green coffee processing and especially roasting will have a variable volatiles composition (Sivetz, 1963). The investigation of volatile pattern of coffee from different geographic origins and species gave different volatile compounds (Freitas and Mosca, 1999).

Green coffee is prepared from the ripe berries of the coffee tree. Two basic methods of processing, dry and wet process are used. Dry processed coffee is generally considered to be lower quality than wet processed and commands a lower price (Clarke, 1985; Varnam and Sutherland, 1994). The wet processing method perhaps the operation difference from the dry process is the fermentation, which step producing desirable flavor compounds during fermentation.

Roasting of coffee beans is thermal process. The characteristic aromas and flavor are developed during roasting as a result of pyrolytic, Maillard reaction and other chemical reactions. The large numbers of different volatile compounds more than 800 difference compounds from wide range chemical classes have been identified in roasted coffee (Mendes, 2001; Schenker et al., 2002). The degree of roasting had an impact on the concentrations of a series of important compounds in roasted beans (Redgwell et al., 2002; Oosterveld et al., 2003). In Thailand, there are a few studies and researches in volatile compounds of coffee although there is cultivation for consume inside the country and export.

Thus, the aim of this study was to investigate the effect of different species, processing procedure and degree of roasting on volatile compounds production in Thai coffee. These experiments were set up in order to compare the method of green coffee processing and drying method between solar drying and tray dryer. If it is not different, it can use tray dryer instead of drying in the solar became sometimes we can not control the solar drying as well as to reduce the drying time.

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CHAPTER 2

LITERATURE REVIEW

Thai coffee

Coffea cultivated in Thailand contains 2 species, *Coffea arabica* and *Coffea canephora* (robusta) (Jirasawat, 2003).

Coffea arabica can be grown in the optimum temperature between 15 to 24^{o}C, cultivated in the northern of Thailand. *C. arabica* is higher quality than robusta and commands a higher price. Furthermore, *C. arabica* also gives good flavor and aroma products.

Coffea canephora (robusta) can be grown in the optimum temperature between 24 to 30 ^{o}C, cultivated in southern of Thailand. Robusta contains higher caffeine than arabica does.

Green coffee processing

Green coffee is prepared from berries of coffee trees by a relatively complex series of process steps carried out entirely within the producing countries. The fundamental purpose of green coffee processing is the recovery of the beans, by removing the various covering layers and drying to produce green beans with a moisture content below 12%. Dry process and wet process, are similar processes including grading, cleaning and polishing are performed (Clarke, 1985; Varnam and Sutherland, 1994).

Dry processing method

In the dry processing, the fruit are allowed to remain on the tree until the fully ripe stage. After harvesting, coffee berries are laid out in the solar to dry until 12% of moisture content in the final beans. The dry method is simpler and cheaper, but the coffee product is usually lower quality than the product from wet processing (Sivetz, 1963; Clarke, 1985; Varnam and Sutherland, 1994). The coffee during the drying period, which lasts 8-10 days in favorable conditions, solar drying is subject to the vagaries of atmospheric conditions, together with the possibilities of growth of both desirable and undesirable microorganisms generating substances from the drying pulp, affecting subsequent flavor of the coffee brew made from the coffee after roasting (Clarke, 1985). Silva et al. (2000) reported of the microbial population associated with dry processing of arabica coffee including 32 species of bacteria, 24 species of yeasts, and 8 species of filamentous fungi. They include groups, which contain many fermentative bacteria and yeasts, cellulolytic bacteria, and pectinolytic bacteria, yeasts and filamentous fungi. Hot air drying is widely used in large scale operations in Brazil and also in Africa. Hot air can be used for the entire drying process, which is reduced in length to 3 days. The time which the coffee is maintained at a given temperature during drying process is just as important in its effect on quality. Overheating during drying produces sour or cooked flavors in the brewed coffee (Sivetz, 1963).

Wet processing method

This process is more sophisticated than the dry process, and by general consent leads to better quality coffee and commands a higher price (Clarke, 1985). Only ripe berries should be used for wet processing, classification by flotation in water is most convenient and involves at least two stages, first to remove stones and dirt, and second to separate cherries. Pulping involves mechanically 'tearing off' the skin and soft pulpy part of the berry. This stage is considered to be the most important stage in wet processing before fermentation. Fermentation procedure is necessary to remove any residual adhering pulp and the mucilaginous layer. If the mucilage remains present during drying, there is the risk of undesirable fermentation, which is detrimental for the quality of the coffee (Clarke, 1985).

Chemical composition of green beans

Caffeine

Caffeine is an important factor in determining the bitter character. The caffeine content of green beans varies according to species. Robusta coffee contains 2.2% on a dry matter basis (dm), and arabica 1.20% dry matter basis (Varnam and

Sutherland, 1994). Roasting process lead to loss of caffeine, but in practice the loss is small (Sivetz, 1963).

Trigonelline

Trigonelline, the N-methylbetaine of pyridine-3-carboxylic acid, is found in all commercial and some wild species. The compound is present in arabica at levels of 1.0% dry matter basis and in robusta at levels of 0.7% dry matter basis. Green bean processing, dewaxing and decaffeination procedures have little effect upon the trigonelline content, but roasting causes progressive destruction (Clifford, 1985; Varnam and Sutherland, 1994).

Chlorogenic acids

The chlorogenic acids (CGA) are ubiquitous in the plant kingdom. They are a family of esters of quinic acid (QA). Analysis of the CGA content is difficult, but it is generally recognized that robusta coffee has a higher content than arabica coffee. Reported levels for robusta are 7.0-10.5% dry matter basis and for arabica is 5-7.5% dry matter basis. The levels of CGA appear to be dependent on species and are unaffected by differences in agronomic practice or method of processing (Varnam and Sutherland, 1994; Flament, 2002).

Proteins and free amino acids

Crude protein contents calculated from total nitrogen contents must be corrected for caffeine and ideally also for trigonelline nitrogen. If such corrections are made there does not seem to be any significant difference between the protein contents of arabica and robusta (approximately 10% dry matter basis), or any significant effect that can be attributed to the method of green coffee processing (Clifford, 1985; Flament, 2002).

Carbohydrates

This is an essential class for the formation of aroma compounds, mainly by caramelization of the low molecular weight sugars and by Maillard reaction with the amino acids. The total amount of carbohydrates represents about 50% dry matter basis of green coffee. The composition is complex with a range of different poly-, oligo- and monosaccharides, subdivided into reducing and non reducing sugars. Polysaccharides are important constituents of green beans and comprise 40-50% dry matter basis. Sucrose is the major free sugar, the quantity present varying according to cultivar, state of maturity, processing applied and storage conditions. Arabica contains 6-8.3% dry matter basis and robusta 3.3-4.1% dry matter basis. Other simple sugars are presented in green beans, including reducing sugars. Quantities are small, total reducing sugar content being 0.1% dry matter basis in arabica and 0.5% dry matter basis in robusta(Clifford, 1985; Varnam and Sutherland, 1994; Flament, 2002).

Lipids

The terms crude and total lipids refer to all material extracted by a specified, usually non polar solvent, and may include non lipid substances such as caffeine (Clifford, 1985). The lipid component of green coffee beans comprises coffee oil, which is primarily presented in the endosperm and coffee wax, which is present on the outer layer. Arabica coffee contains 15% dry matter basis oil and robusta 10% dry matter basis. The oil contains triacylglycerol and considerable proportions of other lipid components. There is no significant difference between arabica and robusta coffee, with respect to fatty acid composition (Varnam and Sutherland, 1994).

Coffee volatiles composition

The volatiles compounds of coffee are largely responsible for the aroma. Green beans are often thought to have no agreeable flavor or aroma, but a large number of volatiles are present. Many of these compounds increase in concentration during roasting, while the concentration of other volatiles falls due to degradation. Aroma of coffee developed during roasting process. Upon roasting, the Maillard reaction, Strecker degradation, pyrolysis, and other chemical reactions produce a large number of different volatiles. More than 800 different compounds from wide range of chemical classes have been identified in roasted coffee (table 2.1) (Varnam and Sutherland, 1994; Flament, 2002; Schenker et al., 2002).

Table 2.1 Volatile compounds of roasted coffee

Volatile compounds	Numbers of Compounds	Volatile compounds	Numbers of Compounds
Hydrocarbons	49	Pyridines	7
Alcohols	19	Quinolines	2
Aldehydes	24	Pyrazines	67
Ketones	83	Qulnoxalines	11
Acids	22	Oxazoles	25
Ester	29	Thiazoles	28
Lactones	7	Thiols	5
Phenols	21	Sulfides	17
Amines	4	Thiophenes	26
Pyrroles	25	Miscellaneous compounds	54
Indoles	3		

Jirasawat (2003)

The furans are found to be the most predominant group of compounds amongst the coffee aroma. They typically have caramel like odors since they are as a result from the pyrolysis of sugars. The pyrazines are the second most abundant class of compounds and contribute to the roasted, walnut cereal, cracker, or toast like flavors in coffee. The pyrroles are responsible for some of sweet, caramel like, and mushroom like aroma in coffee. Conversely, the thiophens are known to have a meaty aroma and are thought to be produced from Maillard reactions between sulfur containing amino acids and sugars. Thiazoles have an even lower presence in the overall aroma, these aroma are formed by sugar degradation (Coffee Research Institute, 2001). Some important characteristic of volatile compounds found in coffee aroma are presented in table 2.2.

Table 2.2 Characteristic of volatile compounds in coffee aroma

Volatile compounds	Aroma quality
Acetic acid	Pungent
4-Methoxy-benzaldehyde	Grass, hay, sweet, mint
2,3-Butanedione	butter
β-Damascenone	fruits, flowers, honey, tea
2,5-Dimethylpyrazine	roasty, nuts
2,6-Dimethylpyrazine	sulfur-like, nuts
2-Ethyl-5-methylpyrazine	musty, burnt
2-Ethyl-3,5-dimethylpyrazine	earthy, roasty, potatoes
4-Ethylguaiacol	flowers, spicy
2-Ethyl-3-methylpyrazine	roasty, nuts
2-Ethyl-5-methylpyrazine	caraway
2-Ethyl-6-methylpyrazine	cheese, caraway
2-Furfurylthiol	roasty, sulfur-like, coffee
Guaiacol	smoky, phenolic, spicy
Hexanal	grass
Furaneol	roasty, sweet, caramel
Methional	potato-like, sweet
2-Methylbutanal	caramel, nuts, malt
3-Methyl-2-buten-1-thiol	green, amine-like
4-Vinylguaiacol	spicy
2,3-Pentanedione	butter
3-Hydroxy-4,5-dimethyl-2(5H)-furanone	seasoning-like

Modified from Coffee Research Institute (2001) and Schenker et al. (2002)

The factors affect the chemical compositions and volatile compounds in coffee

The species of *Coffea*

The two most important varieties of commercial coffee are *Coffea arabica* and *Coffea conephora*, usually known as arabica and robusta, respectively. Commercial coffee beverage is made from arabica or robusta beans or blends of them, the arabica being considered of better quality and is therefore more expensive (Sivetz, 1963; Clarke, 1985; Varnam and Sutherland, 1994).

Martin et al. (1998) investigate of chemical composition between arabica and robusta green coffee by principal component analysis (PCA). The result shown difference in caffeine and total free amino acid between arabica and robusta.

Campa et al. (2004) evaluate of sucrose and trigonelline in arabica and robusta. Contents of these compounds are higher in *Coffea arabica* than in *Coffea canephora* (robusta) green beans.

Ky and others (2001) determined alkaloids, caffeine, trigonelline, chlorlgenic acid and sucrose. Results revealed that the two species showed significant accession differences for all compounds. *C. canephora* diversity was higher than that of *C. arabica*, except for trigonelline and sucrose.

The cultivation of *Coffea*

Coffee beans may be cultivated over a wide geographical area and difference of the weather. The coffee beans may have different chemical and organoleptic properties (Clarke, 1985; Costa et al., 2001). Moreover, similar *Coffea* variety, that from different geographical origins gives difference number of volatile compounds (Jirasawat, 2003)

Freitas and Mosca (1999) investigated the aroma patterns of arabica and robusta from GC-MS and compared using principal component analysis (PCA). Aroma fractions of arabica and robusta give better results in terms of grouping according to the geographical origins.

Processing of green beans

In general, wet process, carefully prepared and handled, is clean in flavor and free from undesirable elements. Dry process, since it is always dried in contact with its mucilage and much more water to remove. It must be dried gently to avoid over drying of the driest portion, the extra layer of the whole fruit offer more resistance to the loss of water. Since the coffee beans from dry process remains moist for much longer period than wet process, the microorganisms, which are always present, have a much better chance to flourish. Especially if rain occurs before it is harvested and produces musty, groundy, or rain damaged flavor (Sivetz, 1963; Varnam and Sutherland, 1994).

Clifford (1985) reported that wet processed arabica gives aromatic with a fine acidity and some astringency, whereas dry process gives less aromatic and less acid but with greater body.

Roasting process

Roasting is the step relating to coffee aroma and flavor development in the processing of green coffee beans. Although the green coffee beans vary in chemical and physical properties, the chemical and physical changes they undergo during roasting are similar even though they vary in degree (Sivetz, 1963). The degree of roasting is qualitatively assessed from color, for simple categorization as a light, medium or dark roast. Roast color will also be broadly correlated with percentage loss of coffee matter, expressed on a dry basis. So that a light roast will show about 3-5% loss, medium 5-8%, dark 8-14% together with the moisture that the green beans will have contained (Clarke, 1985).

Coffee Research Institute (2001) reported that the volatile compositions were developed from roasting process, which affected many chemical reactions (Fig.2.1):

1. Maillard or non-enzymatic browning reaction between nitrogen containing substances, amino acids, proteins, as well as trigonelline, serotonine, and carbohydrates, hydroxy-acids and phenols on the other.
2. Strecker degradation.
3. Degradation of individual amino acids, particularly, sulfur amino acids, hydroxy amino acids, and proline.

4. Degradation of trigonelline.
5. Degradation of sugar.
6. Degradation of phenolic acids, particularly the quinic acid moiety.
7. Minor lipid degradation.
8. Interaction between intermediate decomposition products.

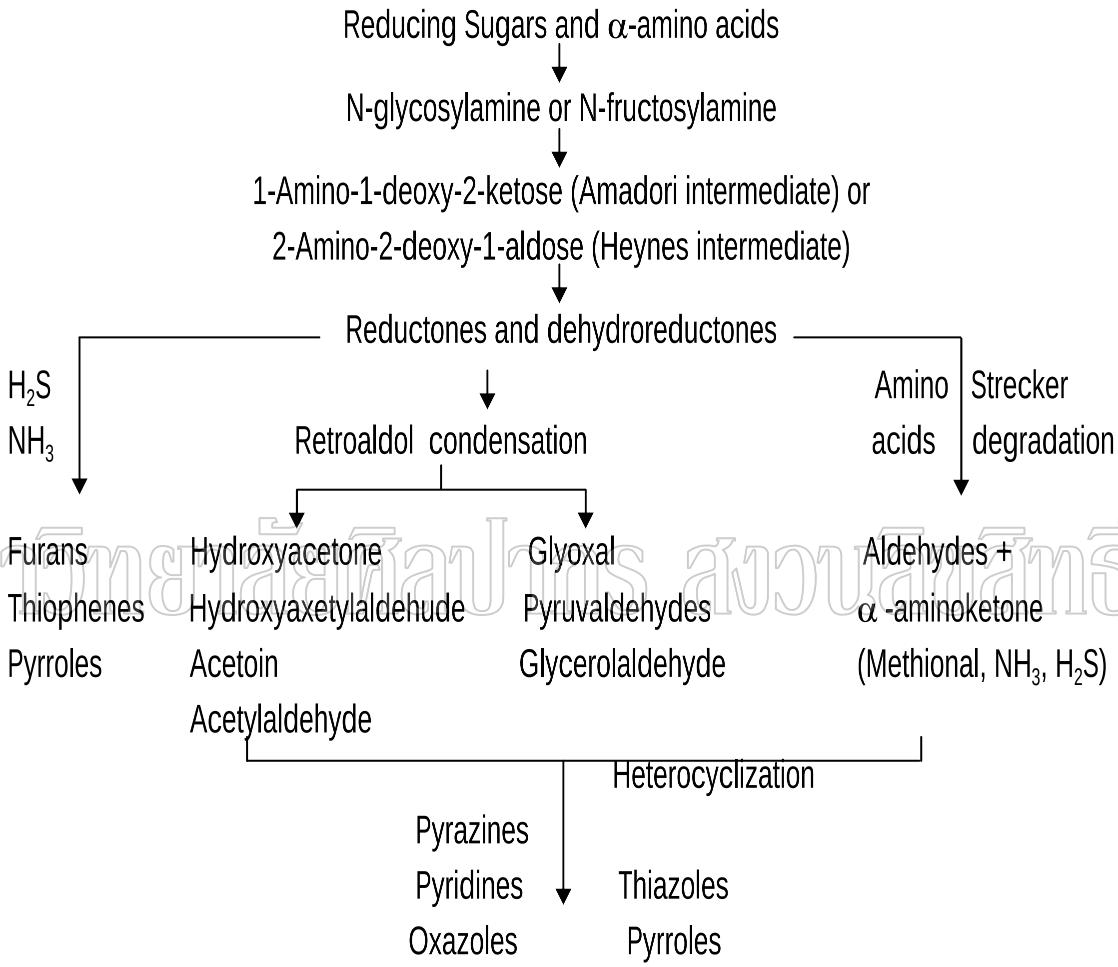

Fig.2.1 Volatile compounds from degradation of carbohydrate and protein composition in coffee beans (Scarpellino and Soukup, 1993)

Maria et al. (1996) determinate roasted arabica coffee by High Resolution Gas Chromatography/Mass Spectrometry (HRGC/MS). It was found that the roasting process was impact on degradation of trigonelline, sucrose, amino acid and arabinogalactan to developed furans, pyrazines and pyridines.

Sharma et al. (2002) investigated the effect of reaction conditions on pyrolysis of chlorogenic acid. The highest weight loss in the tubular reactor for chlorogenic acid was 80% observed at 450°C and above. Although a bulk of pyrolysis of chlorogenic acid was complete below 400 °C, small but significant decomposition was observed at higher temperature.

The carbohydrates presented in coffee beans were reported as a function of the degree of roasting. Oosterveld et al. (2003) reported that oligomers and especially monomers were rapidly converted into Maillard and pyrrolysis products. Cellulose remains unextractable and its solubility was not affected by the degree of roasting. The arabinogalactans were found to be more susceptible to degradation at more severe roasting conditions than the galactans.

The physical changes in the coffee beans during roasting are also technically important. The expansion of the beans including a popping phase, leading to considerably decreased density and increased large microporein in cell wall, which increased extractability in production process of instant coffee (Clarke, 1985; Redgwell et al., 2002).

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CHAPTER 3

INFLUENCE OF GREEN COFFEE PROCESSING AND DEGREE OF ROASTING ON THE CHEMICAL COMPOSITION OF ARABICA COFFEE GROWING IN THAILAND

ABSTRACT

The aim of the chapter was to investigate the effect of different green coffee processing and various degree of roasting (light, medium and dark) on the chemical composition of green Arabica coffee beans. The green Arabica coffee beans were processed by four different methods, dry process and drying with solar dry, dry process and drying with tray dryer, wet process and drying with solar dry and wet process and drying with tray dryer. Then, the chemical compositions, fat content, nitrogen compounds, total sugars and total acidity were determined in both green and roasted coffee beans. The results found that green Arabica coffee beans prepared from different processing showed significant different of fat content and total acidity ($p<0.01$) but they were not significant different ($p>0.01$) of nitrogen compounds and total sugars. It might be due to the fat content and total acidity was generated during green coffee processing whereas nitrogen compounds varied on species and cultivates of coffee. Green beans prepared by the same method (wet process and drying with solar dry) but different degrees of roasting (light, medium or dark) were determined the chemical compositions. The results showed that degrees of roasting resulted in significant different ($p<0.01$) of chemical compositions and may be as a results of chemical reactions changed in roasted beans including degradation of protein, fat, polysaccharide and other components.

Keywords: green coffee processing, roasted coffee, chemical composition, fat contents, nitrogen compounds, total acidity, total sugars

INTRODUCTION

Roasting is and essential step in coffee production for generated aroma, flavor and color of the coffee beans. The mode of heat transfer and the applied temperature profile are the most critical process parameters to have a major impact on the physical and chemical properties of roasted coffee beans (Schenker et al., 2002; Oosterveld et al., 2003). The physical changes in the coffee beans during roasting are also technically important, leading to considerably decreased density and expansion of the beans (Clarke, 1985). The chemical reaction changes include Maillard or non-enzymatic browning reaction, Strecker degradation and degradation of proteins, sugar, polysaccharides and other components.

Degree of roasting is controlled by roasting time and temperature such that they are sufficient for the required chemical reactions to occur, without burning the beans and compromising the flavor of the beverage (Mendes et al. 2001). The degree of roasting is qualitatively assessed from color, for example, simple categorization as a light, medium or dark roast (Clark, 1985).

Thus, the objective of this study was to investigate the effect of different green coffee processing and degree of roasting on chemical composition of roasted Arabica coffee growing in Thailand. These experiments were set up to compare the methods of green beans coffee processing between wet and dry process as well as to compare the drying method of green coffee between solar drying and tray dryer.

MATERIALS AND METHODS

Coffee samples

Coffee cherry varieties of Arabica were obtained from Doi Musor, Tak Province, Thailand. Coffee should be harvested only the red and full like ripe berries. The unripe, overripe or damaged cherries were selected out and then the ripe fruits were cleaned twice with water before further processing.

Green coffee processing

Dry process method 1

Whole berries were placed on cement floor and dried in the solar until the moisture content below 12% (approximately 7 days) and then dried husks were removed from beans.

Dry process method 2

Coffee cherries were dried with Tray dryer (King Machine, Kluay Namtai Kranchang, Thailand). The samples were dried at 40-45^{O}C until the moisture content of the beans were below 12% (approximately 4 days) and then dried husks were removed from beans.

Wet process method 1

The ripe berries were dipped in water to make the soft skin and then the soft ripe berries were put in the pulping machine to remove pulp and mucilage. After that, green beans were separated from the husks and ripe berries. The green beans were put in the plastic bucket (height x diameter, 30 x 15 inches) for fermentation by natural microflora contaminated from green beans or berries. Fermentation of green coffee was done under water at room temperature, this stage should be completed within 36 hours. The green beans were removed from the fermented plastic bags and dried on cement floor under solar dry until the moisture content was 12% or even below (approximately 7 days).

Wet process method 2

The process was done the same as the wet process method 1 but green coffee beans were dried in tray dryer with temperature controlled at 40-45^{O}C until green beans had moisture content at 12% or even below (approximately 3 days).

Roasting process

Seventy gram of green coffee Arabica beans was roasted at 230 ^{O}C by coffee roaster (Precision Coffee Roaster 40201, USA.). The samples were roasted at various degrees, which 7 min for light roasting, 9 min for medium roasting and 12 min for dark roasting. After roasting, the samples were kept in plastic bag (size 11x16 cm) at room temperature for 24 hours to develop flavor in roasted coffee beans. Then the roasted samples were packed in vacuum package. The packaging material was filled in plastic bag (polyethylene, size 18x24 cm). The bags were stored at freezer (approximately -18 ^{O}C) until chemical analysis. The roasted coffee beans were ground into powder by coffee grinding machine (Princess silver, coffee grinder, 2194, USA) before analysis.

Chemical analysis of green beans and roasted beans

Determination of moisture content

The moisture content of green beans and roasted beans were determined by following the procedure of AOAC (1995).

Determination of fat content

The fat content of green beans and roasted beans were analyzed by following the procedure of AOAC (1995).

Determination of nitrogen compounds

The nitrogen compounds were analyzed by following the procedure of AOAC (1995).

Determination of total sugars

The total sugars of green beans and roasted beans were analyzed by following the procedure of Mazzafera (1999).

Determination of total acidity

The total acidity of green beans and roasted beans were analyzed by following the procedure of Mazzafera (1999).

Statistical analysis

All treatments were evaluated in triplicate. Statistic analyses were performed using Randomized Complete Block (RCB) design Experiment to test the effects of different of green coffee processing and various degrees of roasting on chemical compositions. All Data were evaluated using the Statistical Analysis System program (SAS Institute, Inc., 1985). Analysis of variance (ANOVA) was conducted, and the differences between group means were analyzed using the Least Significant Difference (LSD). Statistical significance was established at $p \leq 0.01$.

RESULTS AND DISCUSSIONS

EFFECT OF GREEN COFFEE PROCESSING ON CHEMICAL COMPOSITION

Effect of green coffee processing on chemical composition of green beans

The chemical composition of green beans were investigated and compared between dry and wet process of coffee processing. The results in table 3.1 showed that total nitrogen compounds and sugars found in coffee beans prepared by wet and dry process were not significant differences ($p>0.01$). Therefore, the content of nitrogen and sugars in green beans did not affected by coffee processing. The results were similar to those of Sivetz (1963) and Varnam and Sutherland (1994) who reported that the quantity of nitrogen compounds and sugars in green beans were major important factors by species, environmental and agricultural factors and did not affect by green coffee processing.

The fat content of green bean prepared by dry process and drying with solar dry showed the highest value. This may be as a result of long times of drying (7days) and affect the physical properties of beans. Therefore the long time of drying may be increase efficiency of extraction of fat from green beans.

Total acidity in green beans prepared from dry method was higher than that in wet method. Total acidity was strongly evidence ($p<0.01$) that made significant different between samples prepared from wet and dry process. The high acidity has attributed to fruit fermentation during dry process (Paulo, 1998) whereas wet process, the beans were fermented under water and subsequently the acidic substances released from beans into the water.

In generally, the chemical composition of green beans prepared by drying method between solar dry and tray dryer were not different in term of some chemical compositions, such as nitrogen compounds, total acidity and total sugars (table 3.1) but they were significant different ($p<0.01$) of the fat content.

EFFECT OF GREEN COFFEE PROCESSING ON CHEMICAL COMPOSITION OF ROASTED BEANS

The chemical composition of medium roasted coffee beans were not significant different ($p>0.01$) (table 3.2). So, the results showed that the degree of roasting may be the major factor as affecting the chemical composition more than the green coffee processing (table 3.1). The results were the same as Schenker et al., 2002 and Redgwell et al., 2002.

Effect of degree of roasting on chemical composition

These experiments were set up to investigate the effect of degree of roasting on chemical composition of the selected samples prepared by wet process and drying with solar dry. Moreover, many literatures reported that wet processing gave a higher quality product (Sivetz, 1963; Varnam and Sutherland, 1994). Table 3.3 showed that roasted beans at various degrees of roasting (light medium and dark) affected the chemical compositions of roasted beans. The various degree of roasting gave significant different ($p<0.01$) of moisture content, fat content and total acidity but not significant different ($p>0.01$) of total nitrogen compounds and sugars. The moisture content of the Arabica coffee beans with different degrees of roasting decreased from 11.96 % to 1.60% in light roasted coffee beans, and to 0.76% in dark roasted coffee beans. Nitrogen compounds was constantly at 1.48% in light roasted beans and slightly increased from 1.48 % to 1.62% in medium and dark roasted beans but not significant different. The quantity of nitrogen compounds and were major important factors by species, environmental and agricultural factors (Sivetz, 1963 andVarnam and Sutherland, 1994).

The fat content of coffee beans increased from 0.14% in green beans to 10.37%, 14.58% and 14.75% in light, medium and dark roasted coffee because degree of roasting changed the physical properties of beans. Therefore, the results showed that the more degree of roasting increased the more releasing of fat.

The total acidity of green beans dramatically increased when the beans were roasted. The higher degree of roasting gave the lower total acidity (table 3.3). These results were in agreement with the literature from Varnam and Sutherland (1994) who reported that acetic, formic, citric and malic acid increased in concentration in the early stages of roasting, but were subsequently degraded.

The total sugars of green beans slightly decreased with roasting process, sucrose was rapidly lost (Oosterveld et al., 2002). The carbohydrates of coffee undergo major changes during roasting. The extent of change progressively increased with degrees of roasting from light to dark (Varnam and Sutherland, 1994).

CONCLUSIONS

Different of green coffee processing, wet and dry process, affect the fat content and total acidity ($p<0.01$) but did not affect the nitrogen compounds and total sugars ($p>0.01$). Effect of method of drying process, solar dry and tray dyer, were also investigated and the results showed that the chemical composition were not significant different ($p>0.01$). Therefore, we can use tray dryer instead of solar dry for drying green beans. The tray dryer has more advantage in term of controlling condition as well as decreasing the time of processing. However, we should pay attention in the research about volatile compounds and taste of coffee. Moreover, this experiment also showed that the chemical composition of medium roasted beans from different green coffee processing were not significant different ($p>0.01$).

Effect of degree of roasting on chemical composition of roasted beans from wet process and drying with solar dry decreased moisture content, total acidity, but the fat content increased when increased degree of roasting. The others components as total sugars and nitrogen compounds were not significant different ($p>0.01$).

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Table 3.1 Chemical compositions of green beans

Process	Time of drying (day)	Chemical composition (% dry basis)			
		Nitrogen Compounds	Fat Content	Total Acidity	Total Sugars
Dry [1]	7	1.44 ± 0.05^a	1.63 ± 0.17^a	0.41 ± 0.03^a	0.46 ± 0.04^a
Dry [2]	4	1.41 ± 0.03^a	0.22 ± 0.01^b	0.42 ± 0.03^a	0.47 ± 0.01^a
Wet [1]	7	1.48 ± 0.03^a	0.14 ± 0.02^b	0.25 ± 0.04^b	0.59 ± 0.08^a
Wet [2]	3	1.47 ± 0.04^a	0.13 ± 0.01^b	0.30 ± 0.04^b	0.38 ± 0.05^a

Results are means ± S.D. (n = 3), p<0.01; values of the same column, followed by the same letter (a - c) are not statistically different (p<0.01) as measured by LSD's test.

Dry [1] dry process and drying with solar dry

Dry [2] dry process and drying with tray dryer

Wet [1] wet process and drying with solar dry

Wet [2] wet process and drying with tray dryer

Table 3.2 Chemical components of medium roasted coffee

Process	Chemical composition (% dry basis)				
	Moisture Content	Nitrogen Compounds	Fat Content	Total Acidity	Total Sugars
Dry [1]	1.29 ± 0.07	1.55 ± 0.08	15.64 ± 1.04	4.68 ± 0.78	0.49 ± 0.19
Dry [2]	1.41 ± 0.03	1.50 ± 0.00	13.66 ± 0.48	4.37 ± 0.50	0.40 ± 0.02
Wet [1]	1.18 ± 0.24	1.62 ± 0.10	14.58 ± 1.10	2.95 ± 0.34	0.47 ± 0.09
Wet [2]	1.21 ± 0.10	1.52 ± 0.01	14.09 ± 1.75	3.58 ± 1.23	0.48 ± 0.02

Results are means ± S.D. (n = 3), $p<0.01$; values of the same column are not statistically different ($p<0.01$) as measured by LSD's test.

Dry [1] dry process and drying with solar dry

Dry [2] dry process and drying with tray dryer.

Wet [1] wet process and drying with solar dry

Wet [2] wet process and drying with tray dryer

Table 3.3 Effect of degree of roasting on chemical components of coffee from wet process and drying with tray dryer.

Degree of Roasting	Chemical component (% dry basis)				
	Moisture Content	Nitrogen Compounds	Fat Content	Total Acidity	Total Sugars
Green Bean	11.96 ± 0.14^{a}	1.48 ± 0.03^{a}	0.14 ± 0.02^{c}	0.25 ± 0.04^{d}	0.59 ± 0.08^{a}
Light	1.60 ± 0.23^{b}	1.48 ± 0.03^{a}	10.37 ± 0.79^{b}	5.11 ± 0.19^{a}	0.52 ± 0.05^{a}
Medium	$1.18 \pm 0.24^{b,c}$	1.62 ± 0.10^{a}	14.58 ± 1.10^{a}	2.95 ± 0.34^{b}	0.47 ± 0.09^{a}
Dark	0.76 ± 0.12^{c}	1.62 ± 0.05^{a}	14.75 ± 0.35^{a}	2.29 ± 0.33^{c}	0.40 ± 0.03^{a}

Results are means ± S.D. (n = 3), p<0.01; values of the same column, followed by the same letter (a - d) are not statistically different (p<0.01) as measured by LSD's test.

CHAPTER 4

EFFECT OF SPECIES, GREEN COFFEE PROCESSING AND DEGREE OF ROASTING ON VOLATILE COMPOUNDS OF THAI COFFEE

ABSTRACT

The species affected the volatile compounds of *Coffea arabica* and robusta. Dark roasted arabica had highest amount of furans and pyridines, while robusta contained alcohols, pyrroles and butanoic acid. Green beans prepared in four different processes, dry process and drying with solar dry, dry process and drying with tray dryer, wet process and drying with solar dry and wet process and drying with tray dryer. Only dry and wet processes and drying method with tray dryer were eliminated hexanal and benzaldehyde from green beans while decreased undesirable volatile compounds. After roasting, furans, pyrazines and pyridines were increased as the increase of roasting process from light to medium. At dark roasted degree, the volatile compounds, such as pyrazines, pyrroles and acetic acid were disappeared, whereas 2-methoxyphenol was found. The precursor of volatile compounds, such as acids and sugar decreased as the degree of roasting.

Keywords: arabica, robusta, degree of roasting, green coffee processing, volatile compounds

INTRODUCTION

Aroma is an important attribute to define consumer acceptance of coffee product. Coffee aroma, depending on botanical variety, natural origin, processing history and especially roasting will have a variable volatile composition (Maria et al., 1996; Sivetz, 1963). Green beans are often thought to have no agreeable flavor or aroma before roasting (Varnam and Sutherland, 1994). Flament (2002) reported that many volatile, such as aldehydes, ketones, furans, acetic, propanoic, butanoic acid and sulfur compounds were found in green beans.

Roasting is a time-temperature dependent process, whereby chemical changes are included in the green beans, with a loss of dry mass primarity as gaseous carbon dioxide and other volatile products of the pyrolysis (Clarke, 1988). The maillard reaction, Strecker degradation and other chemical reactions generate most of different volatile compounds. So far more than 800 different volatile compounds identified in roasted coffee (Flament, 2002; Schenker et al., 2002).

Arabica and Robusta coffees have shown significant differences, due to arabica contains lower concentrations of furans, pyrazines, benzene, ketones, and napthalene derivative (2-butanone, 2-heptanone, 3-heptanone, 2-methylpropanol and 2-,3- methylbutanal). Conversely, level of terpenes, 3-methylbutan-1-ol and 2-,3-octanone are higher in arabica than robusta coffee (Varnam and Sutherland, 1994). Arabica is usually the coffee used in blends for the aroma effect and robusta coffee is used for taste and body (Freitas et al., 1999).

The objective of this study was to investigate the effect of different species, green coffee processing and degree of roasting on volatile compounds production.

MATERIAL AND METHODS

Coffee samples

Coffee cherry varieties of Arabica were obtained from Doi Musor , Tak Province, Thailand. Coffee should be harvested only the red and full like ripe berries.

The unripe, overripe or damaged cherries were selected out and then the ripe fruits were cleaned twice with water before further processing.

Green coffee processing

Dry process method 1

Whole berries were placed on cement floor and dried in the solar until the moisture content below 12% (approximately 7 days) and then dried husks were removed from beans.

Dry process method 2

Coffee cherries were dried in tray dryer (King Machine, Kluay Namtai Kranchang, Thailand). The samples were dried at 40-45^{O}C until the moisture content of the beans were below 12% (approximately 4 days) and then dried husks were removed from beans.

Wet process method 1

The ripe berries were dipped in water to make the soft skin and then the soft ripe berries were put in the pulping machine to remove pulp and mucilage. After that, green beans were separated from the husks and ripe berries. The green beans were put in the plastic bucket (height x diameter, 30 x 15 inches) for fermentation by natural microflora contaminated from green beans or berries. Fermentation of green coffee was done under water at room temperature, this stage should be completed within 36 hours. The green beans were removed from the fermented plastic bags and dried on cement floor under solar dry until the moisture content was about 12% or even below (approximately 7 days).

Wet process method 2

The process was done the same as the wet process method 1 but the green coffee beans were dried in tray dryer with temperature controlled at 40-45^{O}C until the green beans had moisture content at 12% or even below (approximately 3 days).

Roasting process

Seventy gram of green coffee Arabica beans was roasted at 230 ^{O}C by coffee roaster (Precision Coffee Roaster 40201, USA.). The samples were roasted at various degrees, which 7 min for light roasting, 9 min for medium roasting and 12 min for dark roasting. After roasting, the samples were kept in sealed plastic bag (size 11x16 cm) at room temperature for 24 hours to develop flavor. Then the roasted samples were packed in vacuum package. The packaging material was filled in plastic bag (polyethylene, size 18x24 cm). The bags were stored in the freezer (approximately -18 ^{O}C) until volatile compounds were analyzed. The roasted coffee beans were ground into powder by coffee grinding (Princess silver, coffee grinder, 2194, USA.) before analysis.

Analysis of volatile compounds by Gas Chromatography-Mass Spectrometry (GC-MS)

Extraction of volatile compounds

Extraction of volatile compounds in ground roasted coffee was done by using Purge & Trap (Tekmar 3100 Sample Conc. Extraction, Tekmar Dohrman). Extraction was repeated twenty of each sample. Volatile compounds were extracted by passing helium gas over the dry samples (0.5 g of ground roasted coffees) contained in sparker of purge and trap system. The samples were heated up to 60 ^{O}C for 2 minutes before started purging. The purge temperature was set at 60 ^{O}C for 10 minutes and flow helium gas through this sample with flow rate 60 ml/min. The extracted volatile compounds were collected in trap (Tenax, Tekmar Dohrman). Then, the volatile compounds were directly desorped and injected to GC-MS. The desorped temperature was set at 200 ^{O}C for 4 minutes and injection time for 1 minute. Blank runs were done routinely, using an empty sample sparker to check the performance of the apparatus.

Gas Chromatography-Mass Spectrometry (GC-MS)

The system comprised HP 5890 gas chromatograph (Agilent Technology Inc.) fitted with Purge and Trap (Tekmar 3100 Sample Conc. Extraction, Tekmar Dohrman) injector with a HP 6890 N mass spectrometer. Helium, as a carrier gas, 2.2 ml/min, was passed through Tenax trap in the Purge and Trap unit and onto the capillary column (HP-5; 30 m long; 0.25 mm internal diameter coated with 0.25 μm thick, Agilent Technology Inc). The volatiles were desorbed from Purge and Trap, and then the chromatography run was started. The temperature of column was programmed starting at 50 ^{O}C hold for 5 min after injection, after which the temperature was increased at rate of 5 ^{O}C/min from 50 ^{O}C to 200 ^{O}C. The total run time was 35 min.

The mass spectrometer was operated in the electron impact mode with ion source temperature, 230 ^{O}C, ionization voltage, 70 eV, Quadupole temperature, 150 ^{O}C and MS interface temperature, 280 ^{O}C. The mass range (m/z) was 40-400 amu.

Identification of volatile compounds

The volatile compounds were identified by comparing their mass spectra with those of known compounds held in the library of the mass spectrometer database, NIST Rev. D.03.00.

RESULT AND DISCUSSIONS

Influence of species on volatile compounds production (Comparision between Arabica and Robusta)

Volatile compounds of *Coffea arabica* and *robusta* from similar dark roasting were determined by purge and trap/GC-MS. The most volatile compounds in *Coffea arabica* were furans, pyridine and 4-pyridinamine. The other volatile compound was

2-methoxyphenol (Table 4.1and Fig.4.1). Wherease, the most volatile compounds were identified in coffee robusta were also furans and pyridines, however lower amount than those in *Coffea arabica*. The other volatile compounds found included alcohols ketone (ethanone, 1-(1-cyclohexen-1-yl)-), pyrroles and butanoic acid (Table 4.1).

Phenolic compound, such as 2-methoxyphenol were found in roasted coffee (Schenker et al., 2002) and increased with degree of roasting (Flament, 2002). Phenolic compounds were generally characteristic of dark roasted and robusta coffees (Varnam and Sutherland, 1994). Jirasawat (2003) reported that the volatile compounds from robusta coffee contributed to the roasty, earthy, spicy and strong odor of sulfur, which the odors caramel, sweet, butter and earthy in arabica coffee.

Effect of green coffee processing on volatile compounds in green beans of *Coffea arabica*

Comparison between dry process and wet process (solar drying)

The experiment was aimed to compare the volatile compounds between dry and wet processing following with solar drying. The highest amounts of volatile compounds presented in green beans prepared from wet processing were aldehydes alkanes furans and acetic acid (Fig.4.2). Moreover, the amount of aldehydes in green beans had lower areas than that prepared from dry process. The aldehydes were identified as hexanal, nonanal, decanal, (E)-2-nonenal and benzaldehyde. The other volatile compounds found in both process included 2-pentylfuran and 2-furanmethanol. However, 3-hydroxy-2-butanone was found only in green beans prepared from dry process (Table 4.2).

Comparision between solar drying and tray dryer

When comparison the volatile compounds between drying method (solar drying and tray dryer), it was found that many volatile compounds in green beans were disappeared, such as hexanal, benzaldehyde, tetradecane and 2-pentylfuran (Table 4.2 and Fig.4.2). The hexanal are formed by oxidation of lipid and gave grassy flavor while benzaldehyde gave stinking flavor in coffee (Flament, 2002;

Schenker et al., 2002). Hexanal and benzaldehyde were undesirable in the products, however, drying method with tray dryer can eliminate both compounds. The volatile compounds in green beans prepared from tray dryer method included 2-furanmethanol and acetic acid was higher than the samples prepared from solar drying method.

Effect of green coffee processing on volatile compounds in medium roasted arabica

Comparision between dry process and wet process (solar drying)

The volatile compounds between dry and wet processing method with solar drying was compared and the results found that the amount of furans and pyridines in coffee beans from wet process was higher than that from dry process, furthermore only 3-ethyl-2-methyl-1,3-hexadiene and 1-(2-furanylmethyl)-1-H-pyrrole were founded in beans prepared from wet process. The main volatile compounds in roasted beans were furans, pyrazines, pyridines and pyrroles. The pyrazines and acetic acid were the highest abundant volatile compounds in coffee beans prepared from dry process, however the alkanes were the highest abundant in beans prepared from wet process (Table 4.3 and Fig.4.3).

Comparison between solar drying and tray dryer

When green coffee beans were dried with tray dryer, some compounds in roasted beans, alkanes and 1-(2-furanylmethyl)-1-H-pyrrole, disappeared. The other volatile compounds found including furans and pyridine were slightly increased, however the amount and abundant of pyrazines was decreased. Acetic acid was not found in roasted beans prepared from wet process and drying with tray dryer. The roasted beans from dry process and drying with tray dryer founded 2,6-dimethyl-2,4-6-octatriene (Table 4.3).

Effect of degrees of roasting on volatile compounds in roasted coffee arabica

Many volatile compounds were identified after roasting of *Coffea arabica* beans and they were different from green beans (Table 4.4). The major volatile compound in roasted beans was furans, pyrazines, pyridines and pyrroles, whereas the most volatile compounds in green beans were aldehydes (hexanal, nonanal, decanal and benzaldehyde) and alkanes (tetradecane and octamethyl-cyclotetrasiloxane) (Table 4.4).

After roasting beans in light, medium and dark degrees, furans and pyridine dramatically increased and being still the most abundant volatile compounds (Fig 4.4). The alkanes and pyrazines were increased from light to medium roasted coffee, however they were was not found in dark roasted beans. The increased degrees of roasting reduced the area of pyrroles and acetic acid until disappeared in dark roasted coffee (Table 4.4 and Fig.4.4). 2-Methoxyphenol was found in dark roasted degree. The other volatile compounds presented in light and medium, respectively were ketones and alkenes (Table 4.4).

Volatile compounds in roasted coffee

Furans

Many furans, such as 2-furanmethanol, 2-furanmethanolacetate, were found in roasted coffee (Freitas and Mosca, 1999). Since green beans contains large quantities of sucrose and other sugars, the furan compounds were formed due to sugars degradation (Maria et al., 1995). When increased degrees of roasting, furans were increased as a consequence of the decreaseing of total sugars (Table3.3 in chapter 3).

Pyrazines

Pyrazine and methyl pyrazine were identified as volatile compounds present in roasted coffee (Maria et al., 1995; Schenker et al., 2002; Varnam and Sutherland, 1994). The pyrazines of roasted coffee are mainly formed by hydroxyl amino acid pyrolysis (Maria et al., 1995; Flament, 2002).

Pyridines

Pyridines are the most important group of volatiles in dark roasted coffee (Varnam and Sutherland, 1994). A high pyridine content is a characteristic feature of highly roasted coffee (Sivetz, 1963). Maria and others (1996) report that pyridines are produced during roasting by the decomposition of trigonelline and hydroxyl amino acid pyrolysis.

Pyrroles

Pyrroles are formed by reactions involving reducing sugar and amino acid, for instance by condensation of glucose and proline or hydroxyproline, so a high degree of sugar is required to form pyrroles (Varnam and Sutherland, 1994; Flament, 2002). The results showed that pyrroles and derivatives of pyrroles were generated increased after roasting (Table 4.4).

Acids

Volatile acids, such as acetic acid were found in roasted coffee, and the maximum concentration was found in light roasting coffee (Coffee Research Institute, 2001). According to the result of total acidity in chapter 3 (Table 3.3), the level of total acidity decreased considerable after roasting. These observations are similar to those of Franca et al. (2004). Furthermore, when increasing degree of roasting, total acidity decreased (Table 3.3). The formation and volatilization of acetic acid due to decomposition of sugars (Flament, 2002).

CONCLUSIONS

The highest volatile compounds in dark roasted arabica coffee were furans and pyridines, whereas robusta roasted of similar degree were alcohols, ketones, pyrroles and butanoic acid.

Green beans prepared from drying method and drying in tray dryer were eliminated the coffee undesirable greeny aroma in coffee products, such as hexanal and benzaldehyde. While these compounds were still found in green beans prepared from drying method and drying in the solar. Therefore, tray dryer can be use in stead of using solar drying because tray dryer can reduce drying times and undesirable volatile compounds, that improved quality of coffee products.

Green coffee processing between dry and wet process with solar drying gave different volatile compounds. The amounts of volatile compounds from in wet process were higher than those in dry process. So, the green coffee processing affected the volatile compounds in green beans and roasted coffee.

Many volatile compounds presented in roasted coffee were furans, pyrazines, pyridines and pyrroles. Pyridines were the highest in dark roasted degree, reversely furans slightly decreased from medium to dark roasted coffee. The other volatile compounds founded were pyrazines, pyrroles and acetic acid and these compounds in light and medium degree disappeared in coffee at dark roasted degree. Degrees of roasting affected the amount and abundant of volatile compounds in coffee products.

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Table 4.1 Volatile compounds of dark roasted coffee arabica and robusta

Compounds	Retention time (min)	Corrected Area*	
		Arabica	Robusta
Alcohols			
2-Methoxyphenol	13.43	1.26E+08	1.19E+09
Mequinol	17.37	-	3.59E+06
1-Naphthalenol	16.75	-	6.24E+06
Aldehydes	-	-	-
Alkanes	-	-	-
Alkenes	-	-	-
Ketones			
Ethanone, 1- (1-cyclohexen-1-yl) -	16.09	-	5.98E+06
Furans			
Furan, 2-methyl-	3.19	-	1.60E+09
Furan, 2-[(methyldithio)methyl]-	16.57	2.09E+07	3.69E+07
Furan, 2-(2-furanylmethyl)-5-methyl-	15.75	1.37E+08	1.92E+08
Furan, 2,2'-methylenebis-	12.41	5.39E+08	-
2-Furanmethanol	8.21	5.90E+09	-
2-Furanmethanol, acetate	10.48	4.40E+09	2.38E+09
Furfuryl pentanoate	16.91	7.96E+06	-
Pyrazines	-	-	-
Pyridines			
Pyridine	5.19	9.49E+09	6.77E+09
4-Pyridinamine	5.78	1.29E+09	-
Pyrroles			
1-H-pyrrole, 1-pentyl-	11.53	-	5.61E+08
1-H-pyrrole, 1-(2-furanylmethyl)-	15.82	-	6.52E+07
Aromatic compounds	-	-	-
Acids and Esters			
Butanoic acid, 3-methyl-,2-furanylmethylester	16.82	-	1.25E+07
Miscellaneous	-	-	-

*Means of corrected area from 10 replication

Table 4.2 Volatile compounds identified in arabica green beans from different green coffee processing

Compounds	Retention time (min)	Corrected Area*			
		Dry process[1]	Dry process[2]	Wet process[1]	Wet process[2]
Alcohols	-	-	-	-	-
Aldehydes					
Hexanal	4.20	2.79E+07	-	2.67E+07	-
Nonanal	12.98	2.62E+07	2.38E+07	1.57E+07	2.88E+07
Decanal	16.14	7.76E+06	4.32E+06	9.05E+06	8.07E+06
2-Nonenal, (E)-	14.80	2.23E+06	-	-	-
Benzaldehyde	8.35	6.49E+06	-	4.39E+06	-
Alkanes					
Tetradecane	21.67	5.49E+06	-	2.38E+06	-
Cyclotetrasiloxane, octamethyl-	9.69		6.50E+06	1.46E+07	-
Alkenes	-	-	-	-	-
Ketones					
2-Butanone, 3-hydroxy-	6.38	2.51E+06	-	-	-
Furans					
Furan, 2-pentyl-	9.32	1.63E+07	-	1.38E+07	-
2-Furanmethanol	8.21	1.29E+06	5.80E+06	4.26E+06	7.29E+07
Pyrazines					
Pyrazine, 2,6-dimethyl-	8.08		-	-	3.66E+07
Pyridines	-	-	-	-	-
Pyrroles	-	-	-	-	-
Aromatic compounds	-	-	-	-	-
Acids and Esters					
Acetic acid	4.01	5.33E+06	2.18E+07	1.50E+07	1.49E+08
Miscellaneous					
Methyl Salicylate	15.77	7.53E+06	-	-	-

*Means of corrected area from 10 replication

Dry [1] dry process and drying with solar dry

Dry [2] dry process and drying with tray dryer.

Wet [1] wet process and drying with solar dry

Wet [2] wet process and drying with tray dryer

Table 4.3 Volatile compounds identified in medium roasted arabica from different green coffee processing

Compounds	Retention time (min)	Corrected Area*			
		Dry process[1]	Dry process[2]	Wet process[1]	Wet process[2]
Alcohols	-	-	-	-	-
Aldehydes	-	-	-	-	-
Alkanes					
Cyclohexane, 1-methyl-4-(1-methylethenyl)-, cis-	12.1	-	-	8.73E+07	-
Eicosane	18.28	3.17E+06	-	-	-
Alkenes					
1,3-Hexadiene, 3-ethyl-2-methyl	12.13	-	-	4.29E+07	9.29E+07
2,4,6-Octatriene, 2,6-dimethyl-	14.12	-	4.00E+07	-	-
Ketones	-	-	-	-	-
Furans					
Furan, 2-[(methyldithio)methyl]-	16.57	-	9.57E+06	6.24E+06	5.53E+06
Furan, 2-(2-furanylmethyl)-5-methyl-	15.75	2.44E+07	6.28E+07	4.46E+07	6.95E+07
Furan, 2,2'-methylenebis-	12.41	3.24E+08	6.93E+08	5.36E+08	6.27E+08
2-Furanmethanol	8.21	-	9.54E+09	8.87E+09	9.44E+09
2-Furanmethanol, acetate	10.48	4.58E+09	6.21E+09	5.61E+09	6.52E+09
2-Furanmethanol, propanoate	13.3	7.99E+07	4.20E+08	9.59E+07	1.82E+08
2-Furancarboxaldehy, 5-methyl-	9.78	-	4.62E+08	4.95E+08	3.67E+08
Pyrazines					
Pyrazine, methyl-	6.05	1.92E+09	2.55E+09	2.04E+09	8.91E+08
Pyrazine, 2,3-dimethyl-	7.51	4.25E+09	-	-	-
Pyrazine, 3-ethyl-2,5-dimethyl-(3,5)	13.19	-	-	1.29E+08	-
Pyridines					
Pyridine	5.19	5.88E+09	6.87E+09	5.83E+09	1.08E+10
4-Pyridinamine	5.78	-	-	4.44E+08	-
N-Vinylpyridinium bromide	4.22	-	-	2.41E+09	-

Table 4.3 (continued) Volatile compounds identified in medium roasted arabica from different green coffee processing

Compounds	Retention time (min)	Corrected Area*			
		Dry process[1]	Dry process[2]	Wet process[1]	Wet process[2]
Pyrroles					
1-H-pyrrole, 1-(2-furanylmethyl)-	15.82	-	-	7.74E+06	-
Aromatic compounds	-	-	-	-	-
Acids and Esters					
Acetic acid	4.01	4.95E+09	6.40E+09	4.21E+09	-
Miscellaneous					
para-Methoxybenzenethiol	15.94	7.39E+06	1.62E+07	9.12E+06	-

*Means of corrected area from 10 replication

Dry [1] dry process and drying with solar dry

Dry [2] dry process and drying with tray dryer.

Wet [1] wet process and drying with solar dry

Wet [2] wet process and drying with tray dryer

Table 4.4 Volatile compounds identified in roasted arabica from wet process with solar drying at different degree of roasting

Compounds	Retention time	Corrected Area*			
		Degree of roasting			
		Green beans	Light	Medium	Dark
Alcohols					
2-Methoxyphenol	13.43	-	-	-	1.26E+08
Aldehydes					
Hexanal	4.20	2.67E+07	-	-	-
Nonanal	12.98	1.57E+07	-	-	-
Decanal	16.14	9.05E+06	-	-	-
Benzaldehyde	8.35	4.39E+06	-	-	-
Alkanes					
Tetradecane	21.67	2.38E+06	-	-	-
Cyclopentasiloxane, decamethyl-	14.72	-	8.07E+06	-	-
Cyclohexane, 1-methyl-4-(1-methylethenyl)-, cis-	12.1	-	-	8.73E+07	-
Cyclotetrasiloxane, octamethyl-	9.69	1.46E+07	-	-	-
Alkenes					
1,3-Hexadiene, 3-ethyl-2-methyl	12.13	-	-	4.29E+07	-
Ketones					
2-Cyclopenten-1-one,3-ethyl-2-hydroxy	13.41	-	3.71E+07	-	-
Furans					
Furan, 2-pentyl-	9.32	1.38E+07	-	-	-
Furan, 2-[(methyldithio)methyl]	16.57	-	5.26E+06	6.24E+06	2.09E+07
Furan, 2-(2-furanylmethyl)-5-methyl-	15.75	-	3.07E+07	4.46E+07	1.37E+08
Furan, 2,2'-methylenebis-	12.41	-	2.95E+08	5.36E+08	5.39E+08
2-Furanmethanol	8.21	4.26E+06	7.19E+09	8.87E+09	5.90E+09
2-Furanmethanol,acetate	10.48	-	3.55E+09	5.61E+09	4.40E+09
2-Furanmethanol, propanoate	13.3	-	-	9.59E+07	7.96E+06
2-Furancarboxaldehyde,5-methyl-	9.78	-	2.99E+09	4.95E+08	-
2,5-Furandione, 3-ethyl-4-methyl-	13.86	-	1.27E+07	-	-

Table 4.4 (continued) Volatile compounds identified in roasted arabica from wet process with solar drying at different degree of roasting

Compounds	Retention time	Corrected Area*			
		Degree of roasting			
		Green beans	Light	Medium	Dark
Pyrazines					
Pyrazine	11.86	-	4.49E+07	-	-
Pyrazine, methyl-	6.05	-	-	2.04E+09	-
Pyrazine, trimethyl	11.42	-	1.47E+08	-	-
Pyrazine,3-ethyl-2,5-dimethyl-	13.19	-	9.79E+07	1.29E+08	-
Pyrazine,2-ethyl-3-methyl	11.15	-	3.69E+08	-	-
Pyridines					
Pyridine	5.19	-	4.16E+09	5.83E+09	9.49E+09
4-Pyridinamine	5.78	-	-	4.44E+08	1.29E+09
N-Vinylpyridinium bromide	4.22	-	-	2.41E+09	-
Pyrroles					
1H-Pyrrole-2-carboxaldehyde, 5-methyl	15.29	-	2.34E+06	-	-
1H-Pyrrole, 2,5-dimethyl-	5.73	-	3.48E+09	-	-
1-H-pyrrole, 1-(2-furanylmethyl)-	15.82	-	1.70E+07	7.74E+06	-
AromaticCompounds					
4-Acetyl-1-methylcyclohexene	14.01	-	1.30E+07	-	-
Benzenamine, 4-methoxy-	11.63	-	4.80E+07	-	-
Acids and Esters					
Acetic acid	4.01	1.50E+07	6.51E+09	4.21E+09	-
Miscellaneous					
para-Methoxybenzenethiol	15.94	-	-	9.12E+06	-
2-(5-Methyl-furan-2-yl)-propionaldehyde	14.57	-	2.52E+06	-	-
1-Buten-3-yne,-4-trimethylsilyl	12.08	-	3.55E+07	-	-
Bicyclo[3.2.2]non-6-en-3-one	14.18	-	8.33E+06	-	-

*Means of corrected area from 10 replication

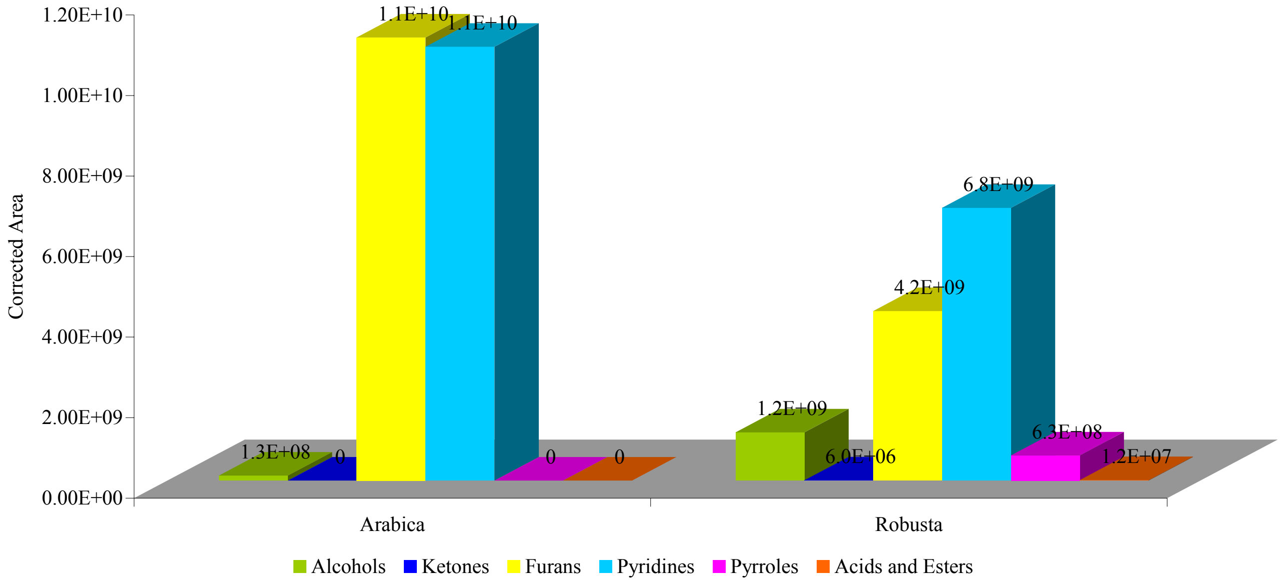

Fig.4.1 Volatile compounds of roasted coffee arabica and robusta

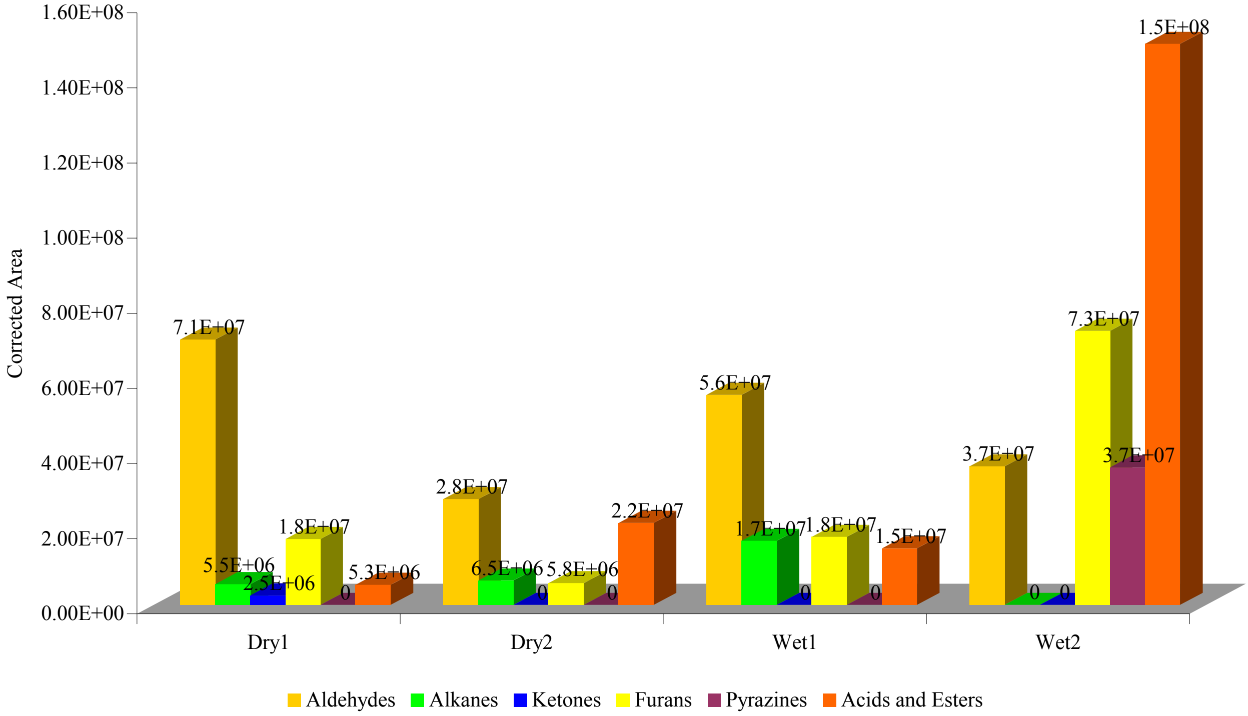

Fig.4.2 Volatile compounds identified in arabica green beans from different green coffee processing

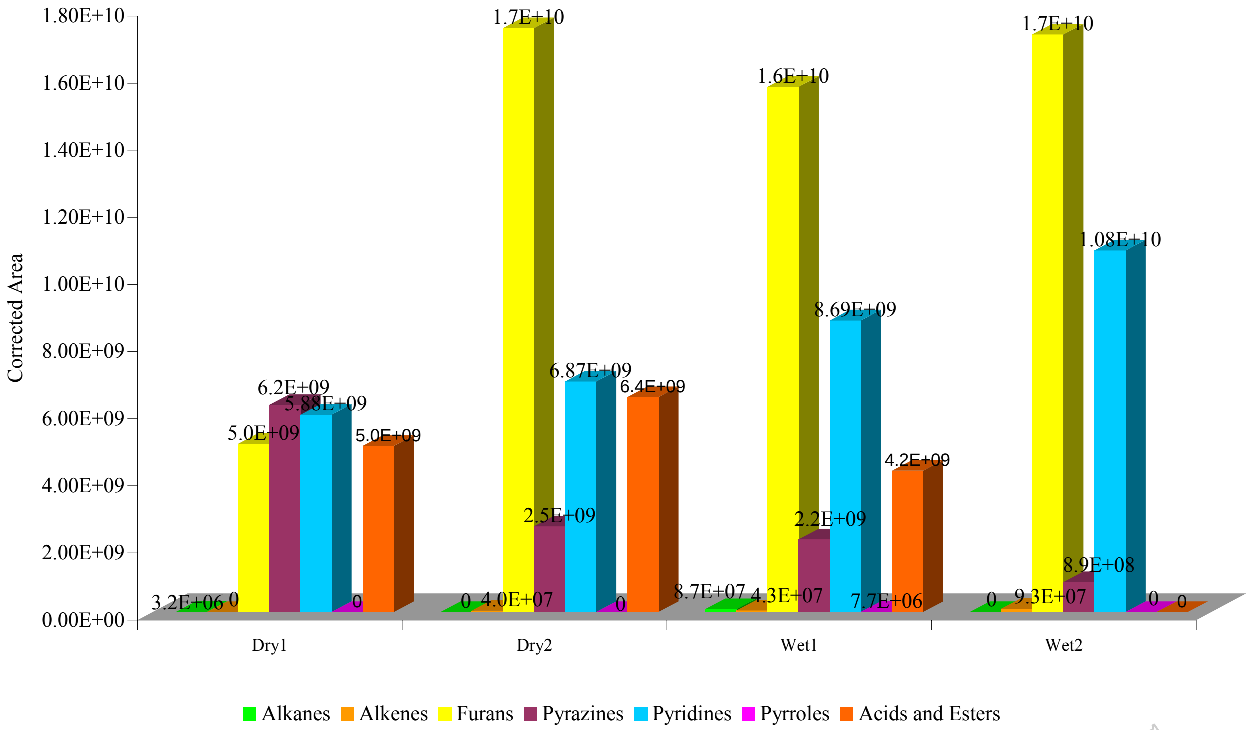

Fig. 4.3 Volatile compounds identified in medium roasted arabica from different green coffee processing

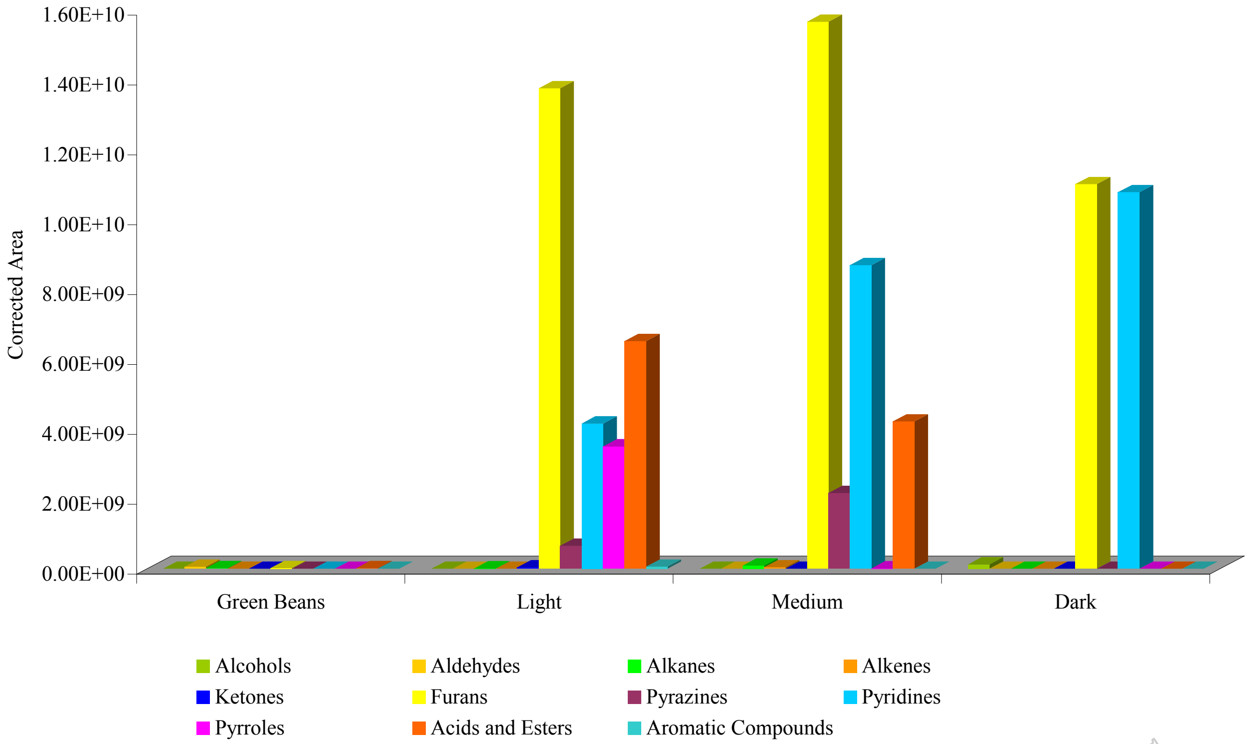

Fig. 4.4 Volatile compounds identified in roasted arabica from wet process with solar drying at different degree of roasting

CHAPTER 5

DISCRIMINATION FOR VOLATILED COMPOUNDS RELEASED FROM ROASTED COFFEE BEANS GROWING IN THAILAND

ABSTRACT

Gas chromatography-mass spectrometry (GC-MS) was employed to analyze and identify a variety of volatile compounds released from two species of coffee beans, the green coffee processing (dry and wet processes) and the elevation of thermal roasting (light, medium, and dark). As a result of the discrimination function analysis (DFA) technique, the unique and different flavor compounds in coffee beans developed from each drying and sequential roasting processes were considerably differentiated. Their quantities and compositions of the coffee flavor substances were ultimately classified, and intimately revealed due to the differences in variety of species, the green coffee process, and the degrees of roasting with reguard to the application of Linear discriminant analysis (LDA). The partial least squares (PLS) plots effectively distinguished the different development of the degradation products and predominantly magnified the extractable aroma changes with an increase in the degree of roasting. As expected, the profiles of the same coffee flavor constituents roasted among the three different degrees of roasting, and two drying procedure not significantly contributed both independent and unique plots and GC-MS profiles.

Keywords: Gas chromatography-Mass spectrometry, arabica, robusta, Linear discriminant analysis, Partial least squares, multivariate analysis

INTRODUCTION

Gas chromatography (GC) is a widely used technique to analyze both quantitative and qualitative constituents in food materials (Teranishi et al., 1999; Gordon and Macrae, 1987). Great improvements in both resolution and sensitivity for the GC utility, in recent years, were tremendously developed with regard to high sensitivity of data acquisition and the use of multivariate statistical-techniques. Unfortunately, the frequently encountered problems presently dealing with some redundant data always lead to require the chemical specialists to interpret the complexity and extract the meaningful information. However, it is always difficult to distinguish between the meaningful information and randomly unworthy variations from the chromatograms (Clifton, 1993).

According to a great variety of volatile compounds detected via GC, discrimination function analysis (DFA) is an alternative and powerful tools presently applied to classify each GC-MS chromatogram profile and then rearrange the entire information in such a way that the useful patterns can be revealed and obsolete from its own originally enormous information (Kemsley, 1998). In general, each peak obtained from individual chromatogram contains its own unique pattern and characteristic, and sequentially must be compatibly identified and then compared with the standard peak to represent the quality and quantity of chemical constituents containing in food materials. PLS techniques are so easy and rapid to distinguish and handle this complexity of an enormous data by setting up the whole data to be subdivided to express the useful meaningful information. Several researchers had employed this technique to achieve the differences in the whole volatile compounds developed due to the differences in various cultivar, the utilization of preservation, or the degrees of fruit ripening for instance, in Roncal cheese (Jesus and Paloma, 2000) and coffee aroma (Maeztu et al., 2001).

The objective of this study was to discriminate the volatile compounds coffee developed from the different species, the various green coffee processing, and the elevated thermal heating of roasting by means of the application of PLS techniques and then compared with GC-MS chromatogram and also evaluate the efficiency of both techniques commonly utilized in food industries.

MATERIAL AND METHODS

GC-MS chromatogram profiles

The volatile chromatogram profiles obtain from GC-MS between the retention times of 0.086 to 35 min were used as the whole fingerprints employed to perform the discrimination between each treatment and then imported all data profiles to Win-DAS program. The chromatogram data from each volatile compounds profiles was individually recorded with respect to the difference in species, the green coffee processes (dry and wet process), the drying procedures (solar and tray dryings), and finally the elevated thermal degrees of roasting. In this experiment, all data were used to classify and distinguish in various treatments composing of 1) the volatile compounds in dark roasted coffee developed from either arabica or robusta, 2) the volatile compounds of medium roasted arabica prepared from solar drying in both dry and wet process, 3) the volatile compounds of medium roasted arabica obtained from different dry process comprising solar drying and tray dryer, 4) the volatile compounds of medium roasted arabica from wet process but different drying methods (between solar drying and tray dryer), and finally 5) the volatile compounds of roasted arabica from wet process with solar drying but applied three different degrees of roasting including light, medium, and dark roasts.

Discriminant Analysis

Win-DAS software program is also used to discriminate each group classification. All data from GC-MS directly at retention time between 0.086 to 35 min imported into Win-DAS. Baseline correct function and area normalization were required to adjust all chromatogram data before calculation and reduce some unwanted and redundant instrumental effects. Subsequently, area-normalization was done to compensate for the overall differences in individual chromatogram response causing by the physical environmental effects, rather than the compositional properties of the releasing volatile compounds (Kemsley, 1998). PLS data compression was performed by the non-orthogonalized formulation and linear discriminant method used for tackling the multiple-group classification. Mahalanobis distance, one of the most widely used parameters in the discriminant function analysis, was applied for the validation (Sivakesava and Irudayaraj, 2001).

RESULT AND DISCUSSION

Influence of species on the volatile compound released between arabica and robusta

Chromatograms data at retention time between 0.086 to 35 min from GC-MS were simultaneously employed to perform the PLS data compression method after the application of baseline correction and area normalization. The PLS plots (between PLS1 and PLS2) of each dark roasted coffee aroma chromatrogram from comprising arabica and robusta prepared by the same coffee processing as show in figure 5.1 extensively clarified and better separated each other in the different plotted regions, The first 2 PLS scores collectively accounted for 67.22% of the total variable. After the application of Linear discriminant analysis (LDA) using the squared Mahalanobis distance to validate each data group, first two PLS provided 100% correct classification of each group samples. Conclusively, it means that the different species of coffee beans were developed the different volatile compounds with regard to the clear presence the chromatograms (Fig5.1), the left plotted was robusta and the opposite was arabica because of arabica and robusta coffees significant differed with respect to robusta coffee normally contained lower concentrations of furans and pyridine. In addition, the other volatile compounds in robusta, such as alcohols, ketone, pyrroles and butanoic acid were still. In contrast, these volatile compounds were not found in arabica (see table 4.1 in chapter 4). Freitas and Mosca (1999) also reported the difference in the volatile compounds consisting of arabica and robusta via the use of Principal component analysis (PCA).

Influence of green coffee processing on volatile compounds in medium roasted arabica

The volatile compounds releasing from dry and wet process using solar drying

Arabica coffee beans obtained from the different green coffee processes were roasted at medium degree to investigate the volatile compounds developed by using different green coffee processing. The same whole fingerprint of GC-MS chromatogram at retention time 0.086 to 35 min were used to discriminate between dry and wet process using solar drying. Significant classification and successful validation of the PLS plots were showed in figure 5.2 and the first two PLS contained about 42.40% of variation. In this case, if the selection of narrow range of volatile profiles at 3-6 min (Fig.5.7) were applied in PLS, the differentiation between dry and wet methods tremendously differentiated with an increase in variation up to 90.30%. According to the previous study, the volatile compounds existed in these region were acetic acid, N-vinylpyridiniumbromide, pyridine, 4-pyridinamine, and methylpyrazine. It represented that the dry and wet processing provide the different in amount and different formation of volatile constituents between dry and wet processing. In addition, the first two PLS scores to provide 95% correction of validation samples for LDA method.

Solar drying process normally utilized a natural microbial fermentation and enzymes to break down the pulp and mucilage, and the appearance of pectinaceous sugars usually produced. Some important chemical substances were composed most of ethanol, acetic, lactic, butyric, propionic, and carboxylic acids (Silva et al., 2000). However, the serious problem was the inability to control drying condition due to its direct dependent upon the environmental conditions. Extended drying periods could result in the growth of microorganisms to adversely affect the flavor of the end products (Varnam and Sutherland, 1994).

In contrast, the wet processing was considerably different due to the removal of fermentation period. It still contained the residual adhering pulp and the mucilaginous layer. The major changes in this process intimately involved the pectin degradation, the hydrolytic enzyme reaction, and the microorganisms growth during fermentation, involving members of the *Enterobacteriaceae*, species of Enterococcus and Lactic acid bacteria (Varnam and Sutherland, 1994), and also the presence of pectinolytic species. During fermentation, the pH deviation was an essential criterion and direct affected the enzyme activity. Normally the pH was about 6.7-6.8 at the beginning and then gradually decreased to 4.2-4.5 at the end of process. It could be explained that the pulped coffee was good and suitable substrate for other enzymes. Moreover, this process was still found some aliphatic acid, acetic, lactic, butyric, and propionic acids (Clarke, 1985).

Comparison between solar drying and conventional tray dryer

The trial tests of green coffee processing to apply the tray drying to decrease the moisture content of green beans to 12% or even below were observed. The PLS plots of chromatograms from solar and tray drying were not different in variety of volatile compounds due to closely of location plots (Fig.5.3 and Fig.5.4). This may be the most beneficial techniques to directly reduce the retention time during drying process and be easy to control by the operator. However, the further investigated was required to confirm about no different in coffee flavor.

Effect of degrees of roasting on volatile compounds in arabica coffee

PLS discriminations of the volatile compounds released from arabica coffee (samples through prepared by wet process with solar drying were conducted to use) at 3 different degrees of roast were observed as shown in figure 5.5. The second PLS score that seem to be the most important for distinguishing each group of samples. The first 2 PLS scores clearly described about 46.46% of the total variance in the whole GC-MS profiles, and this method successfully classified and identified GC chromatograms between the coffee aroma compounds developed during light, medium, and dark roasted with respect to the different plotted location. Subsequently, LDA method was applied to two PLS scores to give 98.28% correct of validation among the tested samples. The chromatograms of the volatile compounds in region

3.5-6 min (Fig.5.8) exhibited more specific characteristics of curve among themselves. The application to PLS plots considerably increased the first 2 PLS scores collectively accounting to 96.74%, showed quality and quantity of the volatile compounds in this region contained more varieties of volatile compound and achieved the better performances. According to GC-MS, the substances found in this region were consisted of acetic acid, N-vinylpyridiniumbromide, pyridine, 1-H-pyrrole,2,5-dimethyl-, 4-pyridinamine and methylpyrazine (see table 4.4 in chapter4). Furthermore, the figure 5.6 shown PLS plots of the light roasted coffee beans and there existed the clear plot on the bottom left and the opposite location to presented the medium and dark roasted coffee beans to closely plotted. The plot of only one PLS score to be importance for presented the pattern of developed of volatile compounds due to increase degrees of roasted.

The use of different heating degree of roasting resulted in the unique development of volatile compounds due to directly impact the formation of great varieties of chemical substances and the concentrations of a series of some important compounds in the roasted coffee beans. Regarding to guaiacol and 2-furfurylthiol normally took place in an unhindered way found in Colombian and Kenyan coffees, and greatly elevated with an increase degree of roasting. In addition, 2,3-butanedione and 2,3-pentanedione developed to a maximum concentration for a medium degree of roasting and exhibited lower concentration in the dark roasted coffee beans (Schenker et al., 2002). Oosterveld and others (2003) investigated the influence of degrees of roasting on the carbohydrate compositions of the roasted arabica coffee beans. The effect of roasting on the sugar compositions of the coffee beans was significant. The relative contents of pectin sugars, arabinose, galacturonic acid, galactose and, to a lesser extent, rhamnose in the coffee bean decreased with an increase of degree of roasting.

As expected, the degree of roasting ultimately impacted with respect to the formation of Maillard's substances including carbohydrates, proteins, lipid, and other chemical compositions. These substances could contribute and develop the different volatile compounds (Flament, 2002).

CONCLUSIONS

The overall detectable peaks received from each GC-MS chromatogram at retention time 0.086 to 35 min were successfully distinguished each others with respect to the different development and formation of some potential released volatile compounds among treatments including the differences in species, green coffee processes, and degrees of roasting. The results from the PLS compression method, and LDA were effectively and successfully classified each group of data up to 90% to 100% of the validation testing. Thus, these techniques may be an alternative tool to investigate and differentiate the flavor development during the processing due to its rapid performance, and no chemistry expertise requirement to describe the differences in the volatile compounds in coffee aroma.

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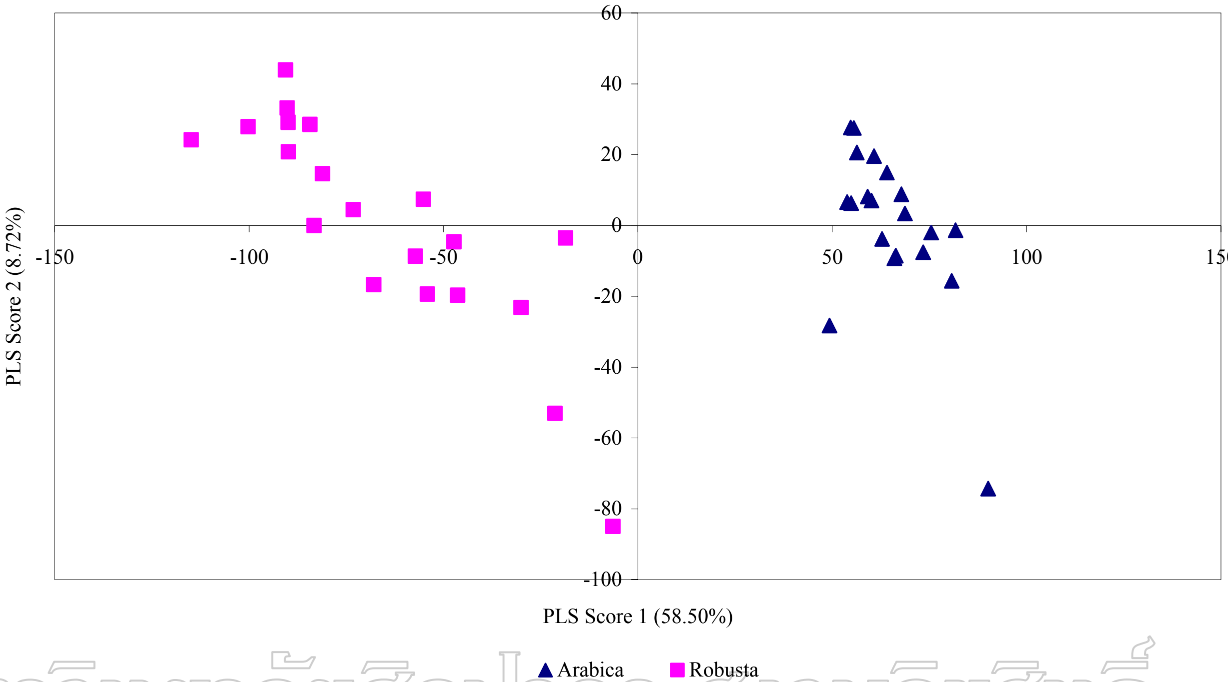


Fig.5.1 PLS plots of flavor profiles of coffee roasted at different species

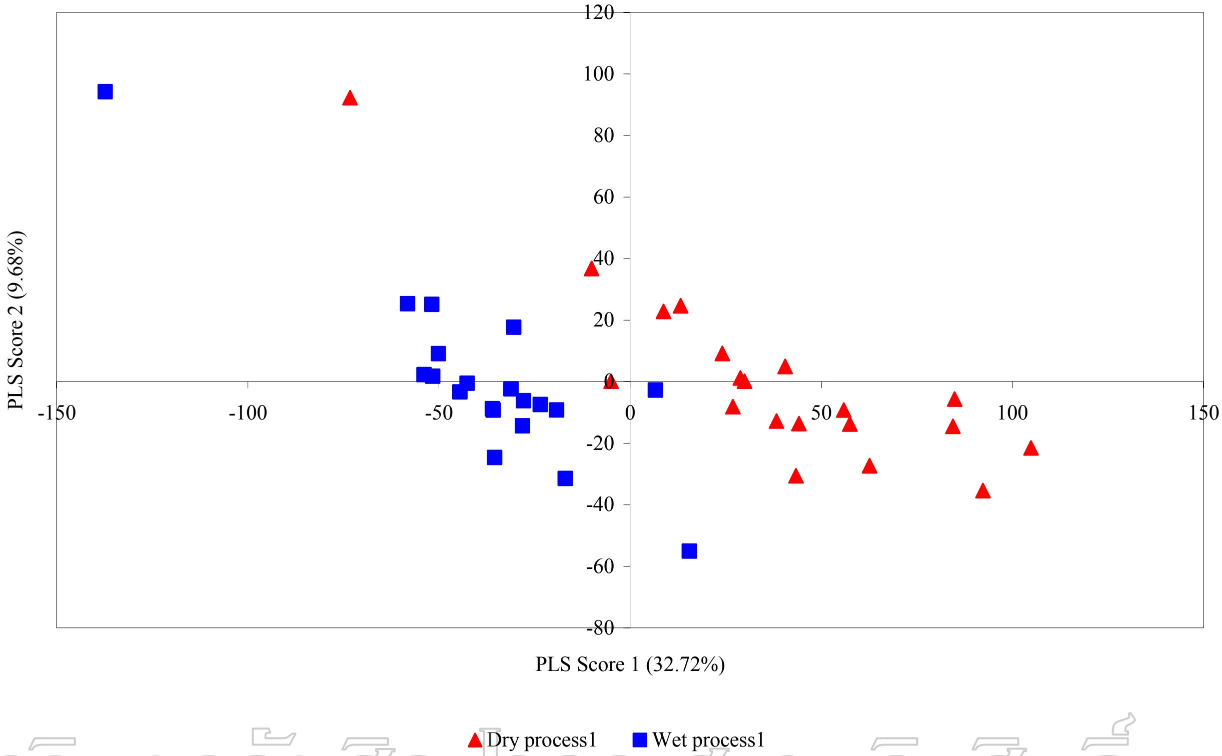


Fig.5.2 PLS plot of flavor profiles of medium roasted arabica prepared from dry and wet process with solar drying

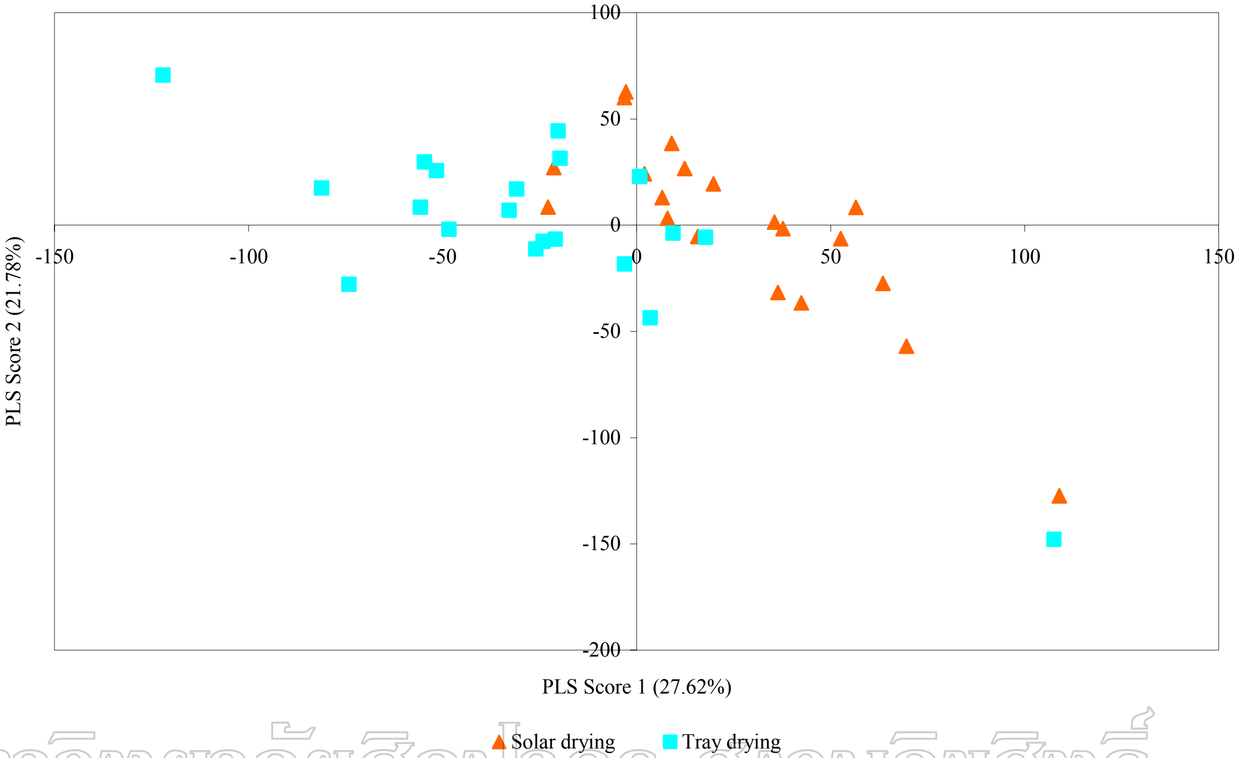


Fig.5.3 PLS plots of volatile profiles of medium roasted arabica from solar and tray drying methods

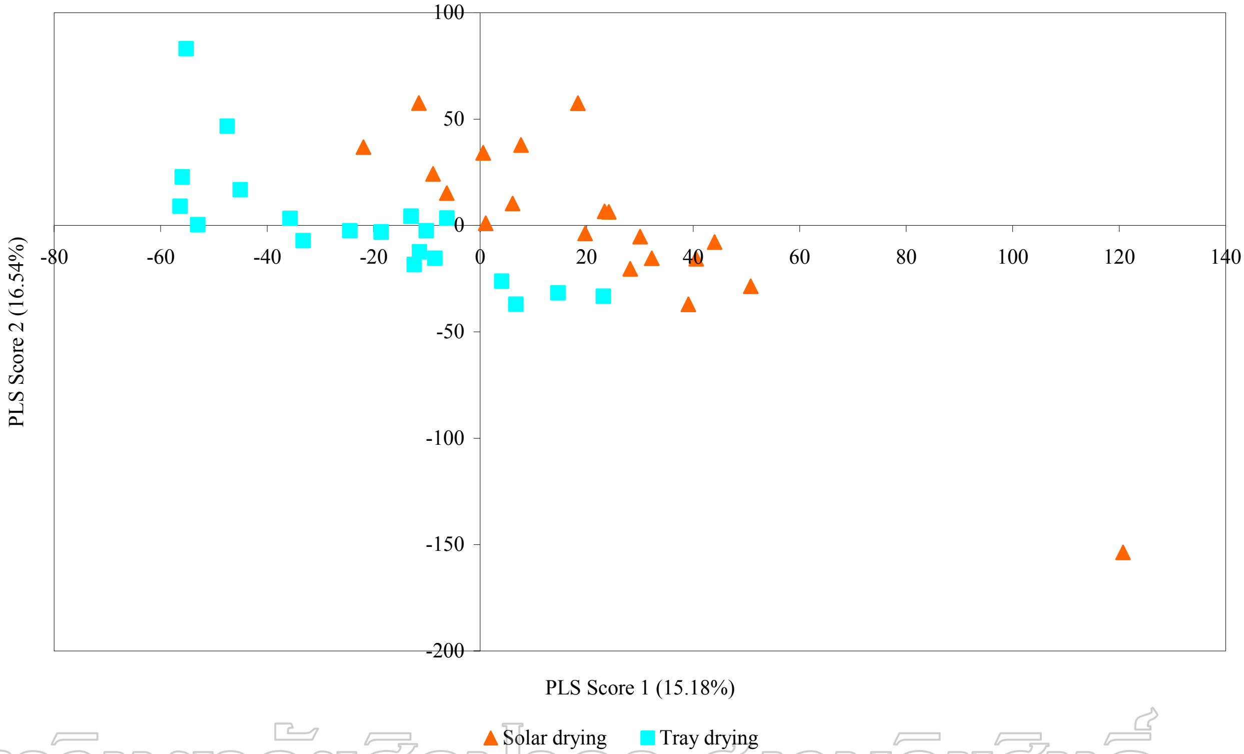


Fig.5.4 PLS plots of volatile profiles of medium roasted arabica prepared from wet process and sequential different solar and tray drying methods

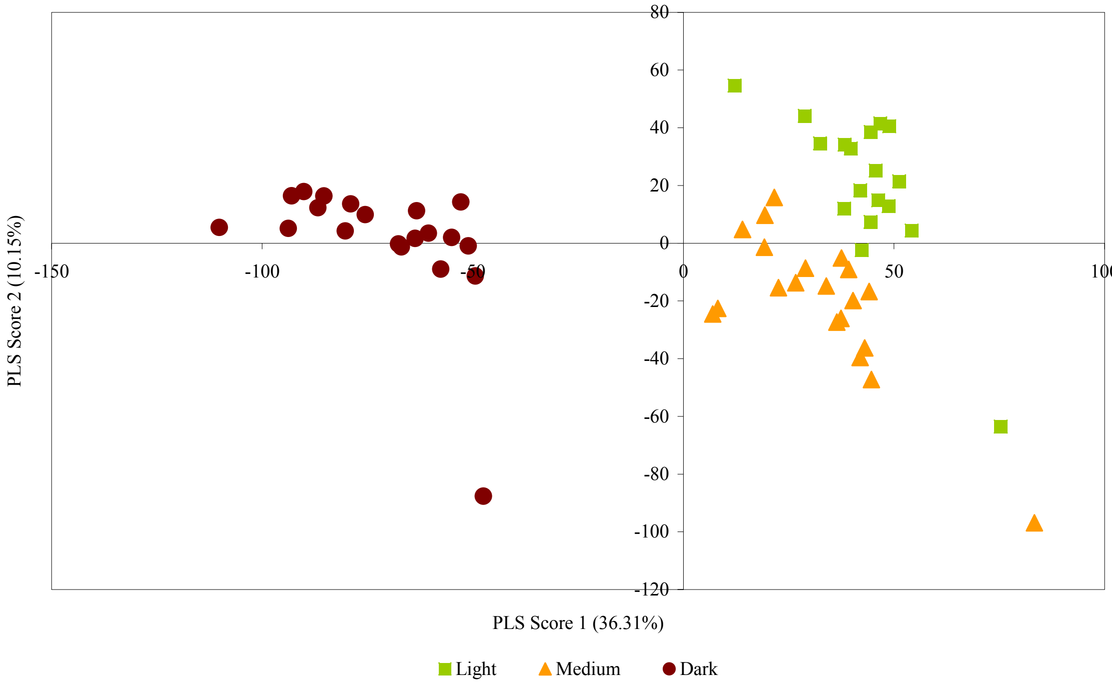


Fig.5.5 PLS plots of flavor profiles at different degrees of roasted arabica

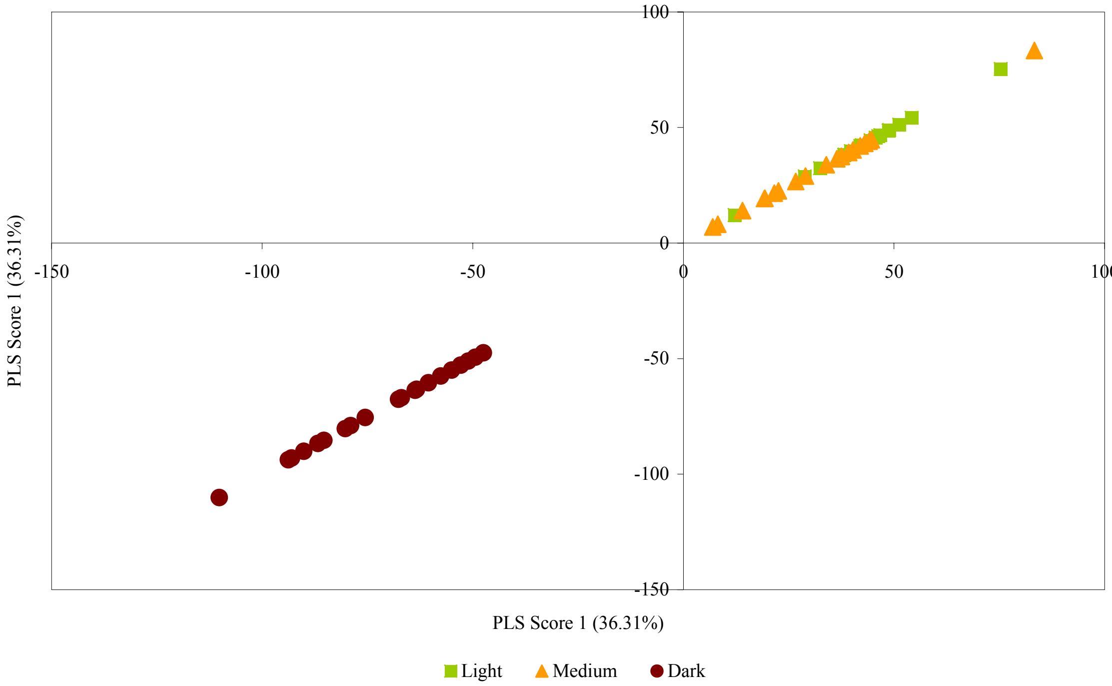


Fig.5.6 PLS plots of flavor profile (PLS1:PLS1) at different degrees of roasted arabica

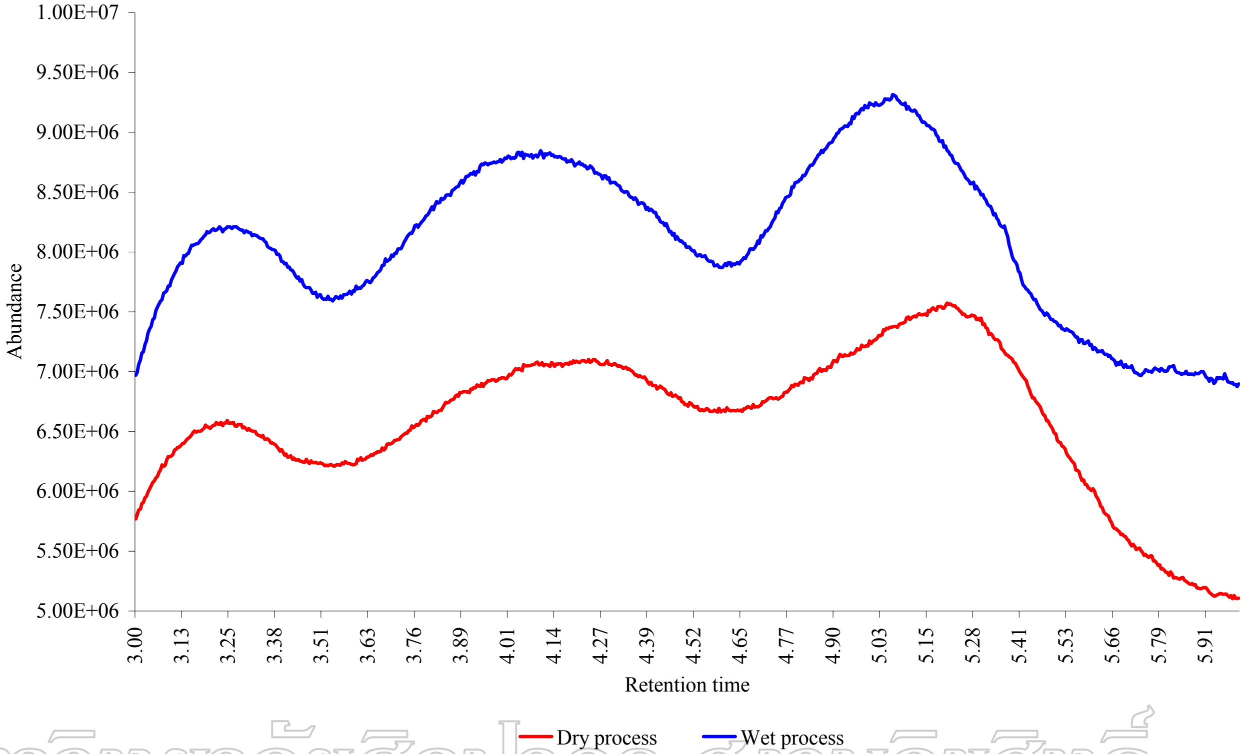


Fig.5.7 Volatile compounds profile from GC-MS (retention time 3-6 min) of medium roasted arabica prepared from dry and wet process with solar drying.

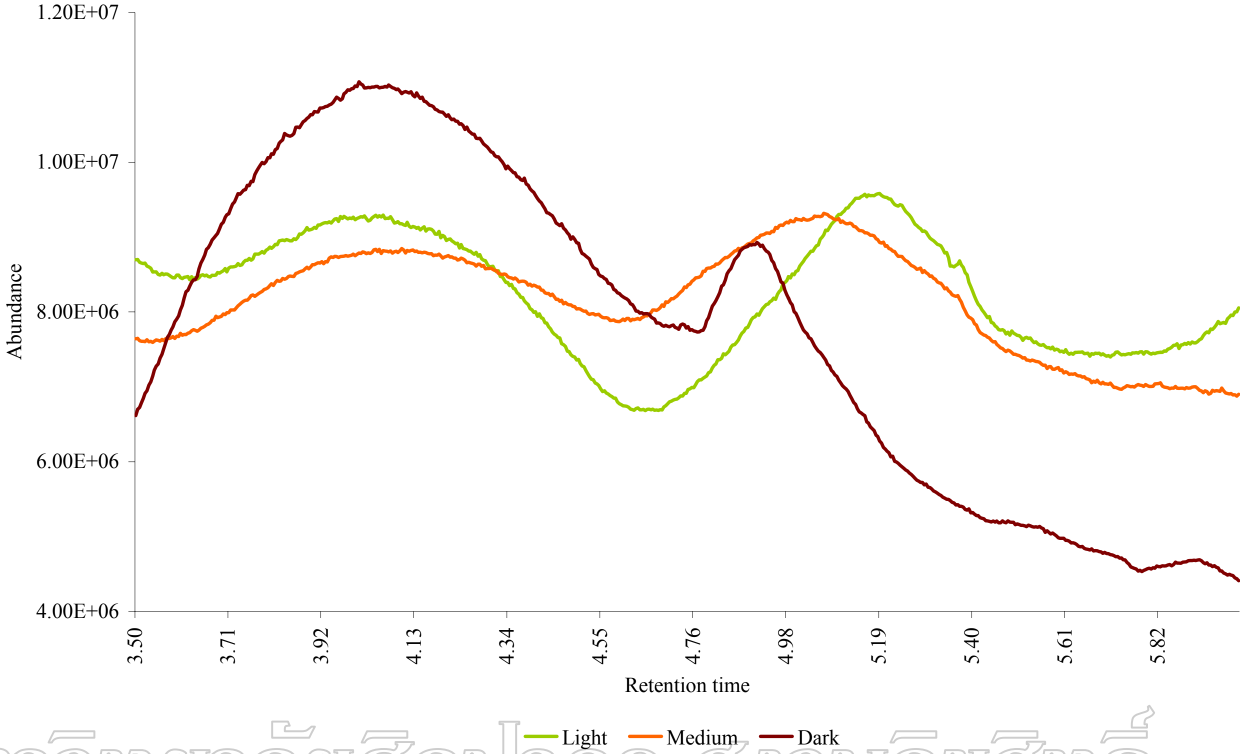


Fig.5.8 Volatile compounds profile from GC-MS (retention time 3.5-6 min) of different degree of roasting arabica prepared from wet process with solar drying

CHAPTER 6

SUMMARY

Influence of species on volatile compounds production (Comparision between roasted arabica and robusta)

The most volatile compounds in *Coffea arabica* were furans, pyridine and 4-pyridinamine. The other volatile compound was 2-methoxyphenol. Whereas, the most volatile compounds identified in coffee robusta were furans and pyridines, however lower amount than in *Coffea arabica*. The other volatile compounds found included alcohols, ketones, pyrroles and butanoic acid.

Effect of green coffee processing on chemical compositions and volatile compounds in green beans of *Coffea arabica*

Comparison between dry process and wet process (solar drying)

The total nitrogen compounds and sugars found in coffee beans prepared by dry and wet process were not significant differences ($p>0.01$). Therefore, the content of nitrogen and sugars in green beans did not affected by the coffee processing. Total acidity in green beans prepared from dry method was higher than that in wet method. Total acidity was strongly evidence ($p<0.01$) that showed the significant different between samples prepared from dry and wet process.

The highest amounts of volatile compounds presented in green beans prepared from wet processing were aldehydes, alkanes, furans and acetic acid. Moreover, the amount of aldehydes in green beans had lower areas than that prepared from dry process. The aldehydes were identified as hexanal, nonanal, decanal, (E)-2-nonenal and benzaldehyde. The other volatile compounds found in both process included 2-pentylfuran and 2-furanmethanol. However, 3-hydroxy-2-butanone was found only in green beans prepared from dry process.

Comparision between solar drying and tray dryer

The chemical composition of green beans prepared by drying method between solar dry and tray dryer were not different in term of some chemical compositions, such as nitrogen compounds, total acidity and total sugars but they were significant different ($p<0.01$) of the fat content.

Volatile compounds in green beans prepared from tray dryer were disappeared, such as hexanal, benzaldehyde, tetradecane and 2-pentylfuran. The volatile compounds in green beans prepared from tray dryer method included 2-furanmethanol and acetic acid were higher than the samples prepared from solar drying method.

Effect of green coffee processing on chemical compositions and volatile compounds in medium roasted arabica

The chemical composition of medium roasted coffee beans prepared from different green coffee processing were not significant different ($p>0.01$).

Comparision between dry process and wet process (solar drying)

The volatile compounds between dry and wet processing method with solar drying was compared and the results found that the amount of furans and pyridines in coffee beans from wet process was higher than those in dry process. The volatile compounds in roasted beans were furans, pyrazines, pyridines and pyrroles. The pyrazines and acetic acid were the highest abundant volatile compounds in coffee beans prepared from dry process, however the alkanes were the highest abundant in beans prepared from wet process.

Comparison between solar drying and tray dryer

Some volatile compounds in roasted beans prepared form tray dryer, alkanes and 1-(2-furanylmethyl)-1-H-pyrrole, disappeared. Generally, the other volatile compounds such as furans and pyridine slightly increased, whereas the amount and of pyrazines decreased. Acetic acid was not found in roasted beans prepared from wet process and drying with tray dryer, but it was found in wet process and drying in the solar.

Effect of degrees of roasting on volatile compounds in roasted coffee arabica

Degrees of roasting (light, medium and dark) affected the chemical compositions of roasted beans. The various degree of roasting gave significant different ($p<0.01$) of moisture content, fat content and total acidity but not significant different ($p>0.01$) of total nitrogen compounds and sugars.

The major volatile compound in roasted beans was furans, pyrazines, pyridines and pyrroles, whereas the most volatile compounds in green beans were aldehydes and alkanes. After roasting beans in light, medium and dark degrees, furans and pyridine dramatically increased and being still the most abundant volatile compounds. The alkanes and pyrazines increased from light to medium roasted coffee, however they were not found in dark roasted beans. The increased degrees of roasting reduced the area of pyrroles and acetic acid until disappeared in dark roasted coffee. 2-Methoxyphenol was found in dark roasted degree. The other volatile compounds presented in light and medium, respectively were ketones and alkenes.

Discrimination function analysis

The differences in the overall peaks from GC-MS chromatograms of roasted coffee flavor compounds were difficult to notify the produce in coffee aroma compounds. PLS technique is so easy and rapid to differentiate and handle the complicate data. The PLS plots presented clearly in the difference of volatile compounds in roasted coffee from differences of species (arabica and robusta), green coffee processing (dry and wet process) and degree of roasting. However, using the whole profile of volatile compounds of medium roasted arabica prepared from different drying method showed no differences in PLS location plots. So, these methods results demonstrated that tray dryer can be used instead of solar drying.

APPENDIX

Chemical analysis of green beans and roasted beans

Green beans were ground with high speed blender and roasted beans were ground with coffee grinder (Princess silver, coffee grinder, 2194, USA.) and then were passed to sieve No.35 before analysis.

Determination of moisture content

The coffee sample were weighed accurately about 2 g with triplicate into moisture can (moisture cans were dried and then cooling in desiccators. Before analysis, the moisture cans were weighed.). The moisture cans and samples were dried at 105^{o}C for 5-6 hours. Then the samples were cooled in desiccators and weighed. Drying was repeat done until the final weight of sample did not alter more than 0.005 g.

Determination of fat content

The round flat bottom flask 250 ml were dried at 105^{o}C for 1 hour and then cooled in desiccators and weighed (repeat until constant weight is achieved). The coffee sample were weighed accurately about 2 g, then wrapped with filter paper and transferred to the extraction thimble of Soxhlet apparatus. The thimble and content were put in the central siphon portion of the Soxhlet apparatus. Then 50 ml of petroleum ether was added into dried 250 ml flask, and then connected with Soxhlet apparatus. The sample was boiled at 140^{o}C for 30 min, then sample was rinsed at similar temperature for 1 hour and evaporated the petroleum ether for 5 min. The flask was dried in an oven at 60^{o}C for 30 min, and then cooled in desiccators and weighed.

Determination of nitrogen compounds

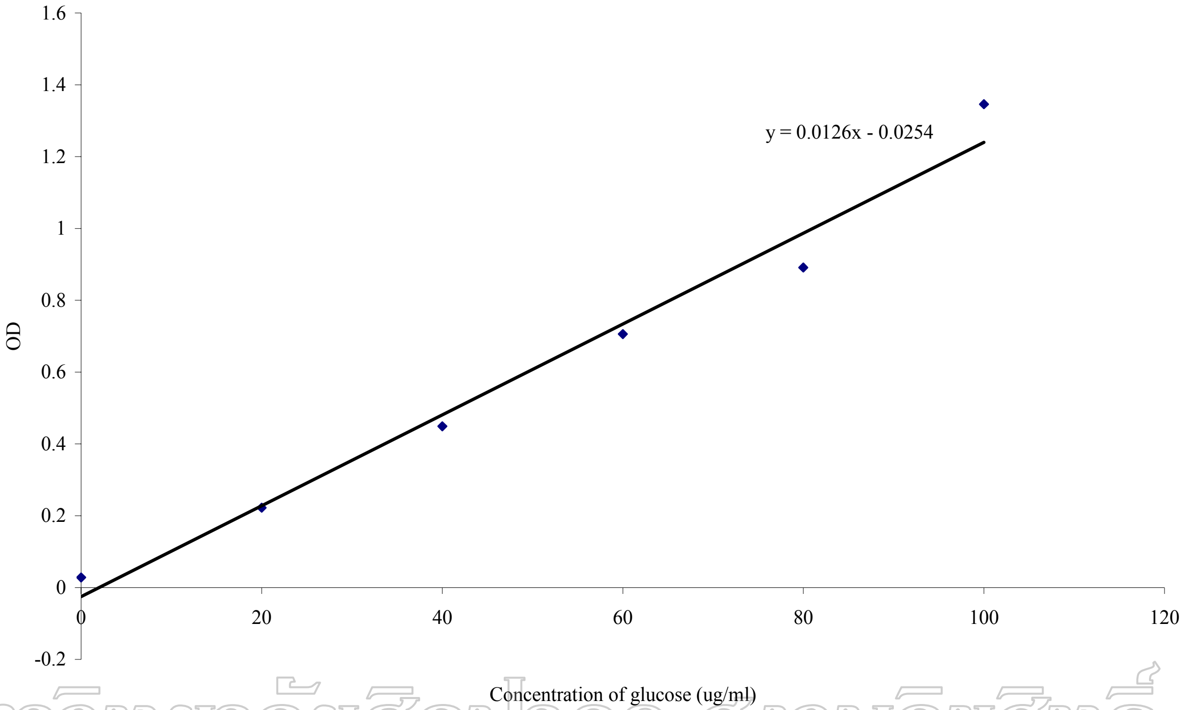
The samples were weighed accurately about 2 g and transferred to digestion tubes. All samples were added with 2.5 g anhydrous $CuSO_4$, 2.5 g of anhydrous K_2SO_4 and 25 ml concentrated H_2SO_4 in digestion tubes and the anti bumping chips were added. The tube was transferred to the preheated digester at 250°C for 30 min, and then increased temperature to 380°C until a clear solution was obtained. After digestion, the tubes were removed from the digester, then cooled and diluted with 140 to 145 ml of distilled water. The sample tube was put in the nitrogen distillation unit. The conical flask containing 25 ml of 4% boric acid and 2 to 3 drops of methyl red were put under the condenser outlet. The alkali solution was dispensed (approximate 60 ml of 50% NaOH) and distilled for 7 min. The ammonium borate solution was formed, and then titrated with 0.1 N HCl to a purplish grey at the end point.

Determination of total sugars

Extraction of total sugars (100 mg of ground coffee in 5 ml of 80% methanol) was performed in a boiling water-bath for 2 hours with occasional agitation. After cooling at room temperature, the extracts were diluted with distilled water to 10 % of methanol. Then, the solutions were analyzed for colorimetric determination of total sugar by Phenol-Sulfuric procedure (Mazzafera, 1999) and pure glucose was used as standard. The standard curve of sugar is shown in figure 7.1

One ml of extracted sample solution was mixed with 1 ml of 5% phenol and 5 ml concentrated H_2SO_4, then agitation and holding at room temperature for 10 min. After holding, the sample tube was put into water controlled temperature at 25°C for 20 min. These sample were determined the optical density (OD) by spectrophotometer at the wavelength 488 nm.

Standard curve of sugar



Determination of total acidity

Ten gram of ground coffee was mixed with 75 ml of 80% ethanol in volumetric flask, stopper and let stand for 16 hours shaking occasionally. These extracts (25 ml of green coffee, 10 ml for roasted coffee) were filtered and diluted to 100 ml with distilled water and titrated with 0.1 M NaOH by using phenolphthalein as pH indicator (Mazzafera, 1999).

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- Poster presentation on The 6th Agro-Industrial Conference (THAIFEX & HALFEX 2004)