Optimisation of extraction conditions of volatile compounds in 'Nam Dok Mai' mangoes

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Abstract: The Thai mango cultivar 'Nam Dok Mai' was extracted using modified Likens-Nickerson's simultaneous distillation-extraction (LNSDE) apparatus. The most appropriate type of solvents and the extraction time were determined. The extraction time varied between 1, 2, 4 and 6 hours. Dichloromethane, hexane and pentane were used as solvents for this experiment. Twenty-nine volatile compounds were identified from 'Nam Dok Mai' mangoes which included aldehydes, alcohols, monoterpene hydrocarbons, sesquiterpene hydrocarbons and non-terpene hydrocarbons. Among the volatile compounds found, *trans*-ocimene; *cis*-ocimene; *β*-elemene; *α*-guaiene; *α*-cubebene; germacrene D; furfural; *(z)*-3-hexen-1-ol; *β*-cubebene and *α*-cadinene were mentioned in previous studies. Dichloromethane was determined as the most efficient solvent in terms of the number and concentration of volatile compounds identified. The 4 hour extraction resulted in a similar number and concentration of the volatile compounds as 6-hour extraction and hence 4 hours was considered as an optimum extraction time.

Keywords: 'Nam Dok Mai' mangoes, extraction, volatile compounds, optimisation

Introduction

Mangifera indica, a member of the family Anacardiaceae, known in English under the common name of mango, is the most popular and celebrated tropical fruit. It is voted number one in the world because of its strong and attractive flavour characteristics. It is grown in almost all tropical countries including Thailand (Andrade *et al*., 2000). Mangoes are an excellent source of vitamin A and phenolic compounds (Hui, 2007). Mango is also one of the most extensively exploited fruits for food, juice, flavour, fragance and colour, making it a common ingredient in new funmctional foods often called super fruits, a term used to highlight potential health value of certain edible fruits (Gross, 2008).

'Nam Dok Mai' is one of the most famous and consumed cultivars in Thailand. It is oval-shaped with a sharp-pointed tip, its ripe fruits are golden-yellow with deep yellow flesh, and the taste is sweet, slightly sour and scented (Thai Mango - Ma-Muang, 2008).

In Thailand, mangoes are used in several Thai dishes. Mangoes in forms of dried and frozen products have become more popular around the globe and therefore the maintenance of the quality is a major concern. Mango flavour is critical to consumer acceptability and an important consideration when the price of the fruit is determined. The economic importance and widespread consumption of the mango fruit have attracted a considerable amount of investigations into its flavour composition (Shang *et al*., 2002).

However, they are seasonal fruit. Thus, many preservation techniques are applied. This may result in some quality changes. Aroma of mangoes is an important quality attribute. This study aims to determine the best extraction conditions for volatile compounds of 'Nam Dok Mai' using Likens-Nickersons' simultaneous distillation extraction (LNSDE) apparatus.

Mango flavour results from the interaction of more than one volatile compound with each other and with sugars and acids (Malundo *et al*., 1996). Creating diverse flavour sensations from a finite number of flavour volatile compounds is feasible by manipulating volatile concentrations. Some volatile compounds are essential contributors to mango flavor (Malundo *et al*., 1996). The major volatile found in mangoes are monoterpenes and sesquiterpene hydrocarbons (Pandit *et al*., 2009). 3-carene gives mango flavor and aroma. *α*-copaene gives mango-like aroma (Occena-Po, 2006).

The study of the volatile fraction has also become necessary and is more than sufficiently justified because these compounds make a major contribution to the consumer's overall perception of the quality of food products. This would help improve its quality and

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therefore would also increase its demand. The volatile fraction of the fruit makes a major contribution with regard to the consumer's decision of acceptance or rejection (Castro *et al.*, 2008).

Aroma compounds are most closely associated with the volatile fraction of food. Identifying and isolating the main volatile compounds in mango can help to improve the quality in artificial fruit drinks/ instant drink mix or in the dried/frozen products. There are several extraction methods that can be used to isolate the volatile compounds, the most popular being solvent extraction, LNSDE, supercritical fluid extraction, static and dynamic headspace. Among them, LNSDE offer the advantage of high extraction efficiency and low cost.

With the existence of LNSDE with its advantages, the aroma volatiles in the Thai mango cultivar 'Nam Dok Mai' will be extracted by the means of modified LNSDE apparatus. The results will be compared with the results of previous work.

The objective of the experiment was to select the most appropriate extraction solvent and the optimum extraction time.

Materials and Methods

Raw materials

A sample of 80 'Nam Dok Mai' no. 4 mangoes grown in Ayutthaya, Thailand was purchased from a supermarket in Bangkok. Samples were washed and peeled before analysis.

Determination of physical and chemical properties

Juice from five mangoes was extracted using a muslin cloth and was used for the determination of titratable acidity, total soluble solids and the pH.

Determination of titratable acidity

A 10 mL volume of juice was titrated with 1 M NaOH using phenolphthalein as the indicator. Titrations were carried on until a colour change was noticed from yellow to orange. The experiment was conducted in triplicate. Percentage of acidity was calculated as shown in equation 1:

$$\text{Titrable acidity (\%)} = \frac{T \times M \times 0.75}{V} \times 100 \qquad (1)$$

Where: T is volume of NaOH (mL), M is molarity of NaOH (M) and V = volume of sample (mL)

Determination of total soluble solids

A small drop of each of the five juice samples was placed onto the handheld refractometer and the total soluble solid content was recorded.

Determination of pH

The pH meter was calibrated in the pH range of 4.0 and 7.0. The electrode was placed into the mango juice samples. The value was recorded once steady.

Determination of colour

Colour of the peel of five mangoes was measured and recorded. The mangoes were then cut into half and the colour of the flesh was then measured and recorded.

Determination of water activity

Approximately 10 g from each of the five mangoes were cut into minute pieces. They were then placed in the water activity crucible and the value was recorded once steady. This experiment was carried out in triplicate.

Determination of moisture content

A sample of 6-10 g from five mangoes was weighed and placed in the vacuum oven at a pressure 88 kPa and at a temperature of 70°C for 24 h. The crucibles were then removed and weighed. The experiment was carried out in triplicates. The weights were noted down and the moisture content calculated using the equation 2:

$$\text{Moisture content in \% on wet basis (MC}_{wb}) = \frac{W_m}{W_p} \qquad (2)$$

Where M_{wb} is wet basis moisture content, W_m is weight of water (kg) and W_p is initial weight of product (kg)

Determination of volatile compounds

Extraction

A modified LNSDE was used. A sample of 250 g (50 g of fruit from five mangoes) was cut and placed into a 2000 mL round bottom flask. A volume of 450 mL of distilled water was added followed by boiling chips. A total of 50 mL of the extracting solvent (pentane/ hexane/ dichloromethane) was added with boiling chips into a 100 mL round bottom flask. Both flasks were placed in heating mantles. The condenser and the flasks were connected together. The acetone condenser was filled to approximately a quarter of its volume followed by dry ice. The cold trap was placed on top of the LNSDE apparatus. Both flasks were heated and the extraction began.

Hexane, pentane and dichloromethane were initially compared on the basis of the extraction time

chosen to be 1 hour. After the most efficient solvent was determined, the time was increased to 2, 4 and 6 hours for that solvent. After the extraction was executed, pear-shaped flasks were used to transfer the sample with the solvent. Sodium anhydrous sulphate was then used to eliminate any remaining water. This was repeated again. The extract was then transferred into an amber screw-cap bottle, wrapped with aluminum foil and kept in the freezer at 4°C.

Concentration

The sample was filtered using filter paper into a pear-shaped flask to eliminate any possible sodium (anhydrous) sulphate remains. An aliquot of 0.1 mL of the standard (0.1% tridecane in dichloromethane) was added to the sample. Nitrogen gas at a controlled pressure was then connected to the pear-shaped flask containing the sample by the means of a pipe and a concentration cap. The sample was concentrated from 50 mL to 1 mL and then transferred into a small brown vial for the identification of the volatile compounds.

Identification

Hewlett Packard (Agilent Technologies) HP-7890A gas chromatograph equipped with an HP 5975C mass-selective detector equipped with an HP-5 MS capillary column (30 m x 0.25 mm i.d. x 0.25 μm of film thickness) from Hewlett Packard were used for all analyses. The temperature program was as follows: start from 40°C for 5 minutes, then rise to 220°C at 10°C/min and held isothermal for 5 minutes. Injector and detector temperatures were 250 and 230°C, respectively. The carrier gas was helium at 1 mL/min flow rate. The mass spectrometer was operated in the electron impact (EI) mode with electron energy of 70 eV and the temperature of ion source of 230°C.

A volume 4 μl of the sample was injected using splitless injection. Compounds were identified by comparing acquired mass spectra with those stored in NIST/NBS mass spectral library. The spectra were printed out and analyzed. Quantification was carried out using the internal standard method.

Results and Discussion

The chemical composition of mangoes in this study was compared to the chemical composition of ripe mangoes published in the handbook of food products manufacturing composed by Hui (2007).

The moisture content range mentioned in the handbook was from 72% to 86%. The moisture content in this experiment was 84%. The water activity was 0.99, which indicated that the mangoes were very juicy and presented a high risk of spoilage.

The maturity indices listed in Table 1 (pH, TSS, and total acidity) was within the range of values specified for mangoes (Hui, 2007). Hence, the fruit used in the experiment could be considered as ripe.

Table 1. Maturity indices of the Thai mango cultivar, 'Nam Dok Mai'

Moisture Content (%wb)	pH	TSS (°Brix)	Total acidity (%)	Water activity	Colour	
					Peel	Flesh
84.00	4.61 (3.8-5.6)*	15.10 (14-23)*	0.17 (0.11-0.48)*	0.99	L 67.5 a 2.52 b 29.5	L 65.4 a 4.98 b 32.1

* Range suggested by Hui (2007)

Selection of solvent for extraction of volatiles from mango

After one hour of extraction, hexane, pentane and dichloromethane revealed 4, 12 and 14 volatile compounds, respectively (Table 2). Hexane extraction produced 3-hexanol, caryophyllene, α-caryophyllene and germacrene D. All of these compounds were also present in the extractions using pentane and dichloromethane, except 3-hexanol. The results in Table 2 show that when compared to pentane and dichloromethane, hexane was clearly out of its league in proving its efficiency of extraction.

Table 2. Comparison of concentration of volatile compounds in Thai mango cultivar 'Nam Dok Mai' (one hour extraction with different solvents)

Volatile compounds	Concentration (ppm)		
	Hexane	Pentane	Dichloromethane
Aldehydes			
2-Hexenal	n.d.	0.03	0.13
Alcohols			
2-Hexanol	n.d.	0.02	n.d.
3-Hexanol	0.37	0.02	n.d.
(Z)-3-Hexenol	n.d.	0.12	0.17
Monoterpene hydrocarbons			
Limonene	n.d.	0.03	n.d.
trans-Ocimene	n.d.	0.03	0.71
cis-Ocimene	n.d.	n.d.	0.15
Sesquiterpene hydrocarbons			
δ-Elemene	n.d.	n.d.	0.01
Copaene	n.d.	n.d.	0.03
Caryophyllene	0.05	0.18	2.81
α-Cubebene	n.d.	n.d.	0.04
α-Guaiene	n.d.	n.d.	0.06
α-Caryophylene	0.03	0.07	1.27
γ-Muurolene	n.d.	0.01	n.d.
Germacrene D	0.04	0.12	1.83
α-Bulnesene	n.d.	0.01	n.d.
α-Cadinene	n.d.	n.d.	0.13
Non-terpene hydrocarbons			
Heptane,2,4-dimethyl-	n.d.	0.04	0.05
Octane,4-methyl-	n.d.	n.d.	0.10

n.d. = not detected

As for hexane, it has a low polarity index of 0.1 and a high boiling point of 68.7ºC (Solvent Properties, 2008). Because of its low polarity index, polar compounds like alcohols, ketone and aldehydes were not extracted. Three hydrocarbons were extracted because hydrocarbons are least polar among these compounds. Due to hexane's high boiling point, many hydrocarbons could not be extracted due their low boiling point. A combination of these factors resulted in hexane extracting the least number of volatile compounds.

Pentane is the only non-polar compound among the three solvents (Solvent Properties, 2008). It has no solvent acidity or basicity. Therefore, it could be expected and it was observed that pentane was effective at extracting non polar compounds like the hydrocarbons. It was not surprising that pentane did not extract high quantities of more polar compounds.

Dichloromethane appears as the most efficient solvent not only because it exhibited the maximum number of volatile compounds but also because it extracted them in a larger concentration than pentane or hexane; for example 2-hexenal cannot be extracted using pentane or hexane but 0.13 ppm of 2-hexenal can be extracted using dichloromethane. Another reason for dichloromethane being found as the most efficient solvent is its ability to extract the volatile compounds normally found in mango which were absent when extracted with pentane but were present in the dichloromethane-extracted sample. Among such compounds are *cis*-ocimene, *δ*-elemene, copaene, *α*-guaiene, *α*-cubebene and *α*-cadinene.

Monoterpene and sesquiterpene hydrocarbons including *cis*-ocimene, *trans*-ocimene, caryophyllene and *α*-caryophyllene had been found to be the major volatile compound in mango (Pandit *et al.*, 2009). When comparing the efficiency of the three solvents in this study, dichloromethane showed the highest concentration of monoterpene and sesquiterpene hydrocarbons followed by pentane and then hexane. Moreover, dichloromethane extracted volatile compounds in higher concentration than the other two solvents.

The efficient extraction by dichloromethane was due to its high polarity and low boiling point of 39.75°C (Solvent Properties, 2008). The polar compounds such as alcohols, ketones and aldehydes but also hydrocarbons with relatively low boiling point could all be extracted with dichloromethane.

Since the highest number of volatile compounds could be extracted with dichloromethane, it could be concluded that this was the most efficient of the three solvents. As a result, further experiments aiming at determining the optimum extraction time were carried out with dichloromethane.

Comparison between different extraction times

The different extraction times are shown in Table 3. The 1-hour, 2-hour, 4-hour and 6-hour extractions using dichloromethane revealed 14, 13, 19 and 18 volatile compounds, respectively.

Table 3. Comparison of different extraction times usingdichloromethane for the Thai mango cultivar 'Nam Dok Mai'

Volatile compounds	Concentration (ppm)			
	1 hour	2 hours	4 hours	6 hours
Aldehydes				
Furfural	n.d.	n.d.	0.36	0.81
2-Hexenal	0.13	0.37	0.08	0.54
(Z)-3-Hexenal	n.d.	0.16	n.d.	0.20
Benzene acetaldehyde	n.d.	n.d.	n.d.	0.01
Alcohols				
(Z)-3-Hexen-1-ol	0.17	2.80	0.78	1.97
3-Hexanol	n.d.	0.08	n.d.	0.05
Monoterpene hydrocarbons				
trans-Ocimene	0.71	0.34	0.73	0.73
cis-Ocimene	0.15	0.04	0.14	0.11
cis-Linaloloxide	n.d.	n.d.	0.01	n.d.
δ -2-Carene	n.d.	n.d.	0.08	n.d.
Limonene	n.d.	tr	n.d	n.d
Sesquiterpene hydrocarbons				
δ -Elemene	0.01	tr	n.d.	n.d.
Copaene	0.03	0.02	0.16	0.01
α-Bourbonene	n.d.	n.d.	0.01	n.d.
β-Bourbonene	n.d.	n.d.	0.04	n.d.
β-Elemene	n.d.	n.d.	0.10	n.d.
Caryophyllene	2.81	1.22	4.50	1.94
β-Cubebene	n.d.	n.d.	0.02	n.d.
α-Guaiene	0.06	tr	0.44	0.08
α-Caryophylene	1.27	0.54	1.82	0.88
α-Cubebene	0.04	tr	n.d.	0.04
γ-Muurolene	n.d.	0.04	0.15	0.02
Germacrene D	1.83	0.53	3.79	1.04
α-Bulnesene	n.d.	0.12	0.34	0.19
allo-Aromadendrene	n.d.	n.d.	n.d.	0.05
α-Cadinene	0.13	0.15	0.46	0.33
Non-terpene hydrocarbons				
Heptane,2,4-dimethyl-	0.05	n.d.	n.d.	n.d.
Octane,4-methyl-	0.10	n.d.	n.d.	n.d.

n.d. = not detected
tr = trace (<0.01 ppm)

Considering the number of the volatile compounds extracted using different extraction times, there is no significant difference between 1 and 2 hours extraction or between 4 and 6 hours at the significance level of 0.05. However, there are some differences between 1 h and 2 h extraction. The 1-hour extractions produced two compounds namely heptane,2,4-dimethyl and

octane,4-methyl that were not found in any other extraction. The 2-hour extraction did not single out any compound that wasn't present after other extraction times.

The 4-hour and 6-hour extraction results differ to some extent as the compounds found after the 4-hour extraction were of higher concentration. The compounds that were not shown after 4-hour extraction but were found after 6-hour extraction were 3-hexanol, *(z)*-3-hexenal, benzene acetaldehyde, *α*-cubebene and *allo*-aromadendrene. Some of these volatile compounds were heat generated volatile compounds e.g. benzene acetaldehyde. Likewise, there were volatile compounds that were found after 4-hour extraction but were absent after 6-hour extraction. These include *cis*-linaloloxide, *δ*-2-carene, *α*-bourbonene, *β*-bourbonene, *β*-elemene and *β*-cubebene. The volatile compounds that were lost after 6-hour extraction may result from the oxidation reactions and decomposition that increase with increasing extraction time. This is a likely reason why *δ*-2-carene, *α*-bourbonene, *β*-bourbonene, *β*-elemene were only found after 4-hour extraction.

Comparing the main monoterpene and sesquiterpene hydrocarbons, it appears that the 4-hour extraction results in the highest concentration in all of them, including *cis*-ocimene, *trans*-ocimene, copaene, caryophyllene, *α*-caryophyllene, germacrene D and *α*-cadinene.

Therefore, it can be concluded that the 4-hour extraction was the most efficient in comparison with other extraction times. Hence, dichloromethane and 4-hour extraction time are considered as the optimum extraction conditions to extract volatile compounds from the 'Nam Dok Mai' mango.

Comparison of current results with previous studies

A total of 29 volatile compounds were identified in 'Nam Dok Mai' mangoes, when considering different solvents and extraction times (Table 4). They include 4 aldehydes, 3 alcohols, 5 monoterpene hydrocarbons, 15 sesquiterpene hydrocarbons and 2 non-terpene hydrocarbons.

Comparing all the volatile compounds found in this study, see Table 4, with previous studies by Tamura *et al*. (2000) and Chitsamphandhvej (2007), 6 volatile compounds were found to be common namely *trans*-ocimene, *cis*-ocimene, *β*-elemene, *α*-guaiene, *α*-cubebene and germacrene D. Overall, 30, 44 and 25 volatile compounds were found in the current study, that of Tamura *et al*. (2000) and of Chitsamphandhvej (2007), respectively.

The major volatiles found in this study were caryophyllene, *α*-caryophyllene and germacrene

Table 4. Comparison between volatile compounds identified in the Thai mango cultivar, 'Nam Dok Mai' in the current and the previous studies

Volatile compounds	Identified in the current study	Identified in previous studies	
		Reference[1]	Reference[2]
Aldehydes			
Acetaldehyde	-	+	-
Benzene acetaldehyde	+	-	-
2-Hexenal	+	-	-
(Z)-3-Hexenal	+	-	-
(E)-2-Hexenal	-	+	-
(E,Z)-2,6-Nonadienal	-	+	-
Furfural	+	+	-
Alcohols			
Ethanol	-	+	-
1-Butanol	-	+	-
Pent-1-en-3-ol	-	+	-
2-Hexanol	+	-	-
3-Hexanol	+	-	-
(Z)-3-Hexen-1-ol	+	+	-
3-Methylbut-2-en-1-ol	-	+	-
1-Hexanol	-	+	-
1-Octanol	-	+	-
1-Hexadecanol	-	+	-
Monoterpene hydrocarbons			
Myrcene	-	+	+
Limonene	+	-	-
Sabinene	-	-	+
trans-Ocimene	+	+	+
cis-Ocimene	+	+	+
allo-Ocimene	-	-	+
cis-Linaloloxide	+	-	-
δ-2-Carene	+	-	-
Sesquiterpene hydrocarbons			
δ-Elemene	+	-	-
Copaene	+	-	-
α-Copaene	-	+	+
α-Bourbonene	+	-	-
β-Bourbonene	+	-	-
β-Elemene	+	+	+
Caryophyllene	+	-	-
β-Cubebene	+	-	+
α-Gurjunene	-	-	+
α-Guaiene	+	+	+
δ-Guaiene	-	-	+
α-Caryophyllene	+	-	-
β-Caryophyllene	-	+	-
trans-Caryophyllene	-	-	+
α-Cubebene	+	+	-
α-Muurolene	-	-	+
γ-Muurolene	+	-	-
(E)-β -Farnesene	-	+	-
Humulene	-	+	-
α-Humulene	-	-	+
β-Chamigrene	-	+	-
β-Selinene+ *α*-Selinene	-	+	+
α-Amorphene	-	-	+
Germacrene D	+	+	+
α-Bulnesene	+	-	-
Aromadendrene	-	-	+
allo-Aromadendrene	+	-	-
α-Cadinene	+	-	+
δ -Cadinene	-	+	+
γ-Cadinene	-	-	+
Cadina-1,4-diene	-	-	+
Velencene	-	-	+
Germacrene B	-	-	+
Calamenene	-	+	-
Caryophyllene oxide	-	+	-
Ketones			
Acetone	-	+	-
2,3-Pentandione	-	+	-
Pent-3-en-2-one	-	+	-
Non-terpene hydrocarbons			
Octane,4-methyl-	+	-	-
Heptane,2,4-dimethyl-	+	-	-
Heptadecane	-	+	-
Esters			
Ethyl acetate	-	+	-
Ethyl formate	-	+	-
Octyl butyrate	-	+	-
Methyl dodecanoate	-	+	-
Ethyl dodecanoate	-	+	-
Methyl tetradecanoate	-	+	-
Ethyl tetradecanoate	-	+	-
Ethyl hexadecanoate	-	+	-
Acetic acid	-	+	-
Butyric acid	-	+	-
Hexanoic acid	-	+	-
Octanoic acid	-	+	-
Decanoic acid	-	+	-
Decanoic acid	-	+	-

+ detected
- not detected
[1] Chitsamphandhvej, 2007
[2] Tamura *et al*., 2000

D. In contrast, *cis*-ocimene, *trans*-caryophyllene, *trans*-ocimene and *α*-humulene were prevalent

in the study done by Chitsamphandhvej (2007). Tamura *et al*. (2000) reported *β*-caryophyllene as the major component. Different isomers of the same compound have been found. For example, caryophyllene, *α*-caryophyllene, *β*-caryophyllene and trans- caryophyllene are 4 isomers of the same compound.

Tamura *et al*., (2000) reported furfural and *(Z)*-3-Hexen-1-ol which were also found in the current study. *β*-cubebene and *α*-cadinene were found in this study and in that by Chitsamphandhvej (2007).

Myrcene, *α*-copaene, *β*-selinene, *α*-selinene and *δ*-cadinene are present in both previous studies but were not found in the current study.

Chitsamphandhvej (2007) uses headspace solid-phase microextraction (HS-SPME) whereas this study uses LNSDE for all the extractions. The simultaneous distillation extraction (SDE) and solvent extraction used by Tamura *et al*., 2000 also are different from the method used in this experiment even though the SDE apparatus used was similar to LNSDE. The difference in volatile compounds and their concentrations could be due to the difference in techniques used for extraction. An additional factor could also be the difference in the maturity indices leading to some volatile compounds being unavailable at a particular stage of fruit maturity.

Conclusions

Twenty-nine volatile compounds from 'Nam Dok Mai' mangoes were identified. These include 4 aldehydes (furfural, 2-hexenal, *(z)*-3-hexenal and benzene acetaldehyde), 3 alcohols (2-hexanol, 3-hexanol and *(z)*-3-hexen-1-ol), 5 monoterpene hydrocarbons (limonene, *trans*-ocimene, *cis*-ocimene, *cis*-linaloloxide and *δ*-2-carene), 15 sesquiterpene hydrocarbons (copaene, *δ*-elemene, *α*-bourbonene, *β*-bourbonene, *α*-cadinene, caryophyllene, *β*-cubebene, *γ*-muurolene, germacrene D, *α*-bulnesene, *α*-guaiene, *α*-caryophylene, *α*-cubebene, *allo*-aromadendrene and *β*-elemene) and 2 non-terpene hydrocarbons (2,4-dimethyl heptane and 4-methyloctane). *(Z)*-3-hexen-1-ol, caryophyllene, *α*-caryophylene and germacrene D are found to be the major volatile compounds of 'Nam Dok Mai' mangoes in this experiment.

Dichloromethane was found to be the most efficient solvent when compared to pentane and hexane, due to higher number and concentration of volatile compounds identified. The 4-hour extraction was found to be the optimum extraction time, as it resulted in the highest number and concentration of the volatile compounds amongst all extraction times

investigated. Comparing previous studies with the current one, the following volatile compounds were found in common: *trans*-ocimene; *cis*-ocimene; *β*-elemene; *α*-guaiene; *α*-cubebene; germacrene D; furfural; *(z)*-3-hexen-1-ol; *β*-cubebene and *α*-cadinene.

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