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# Stability of Essential Oils in Pang-Rum by Gas Chromatography-Mass Spectrometry

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***Abstract*—**Ancient Thai perfumed powder was used as a fragrance for clothing, food, and the body. Plant-based natural Thai perfume products are known as Pang-Rum. The objective of this study was to evaluate the stability of essential oils after six months of incubation. The chemical compositions were determined by gas chromatography-mass spectrometry (GC-MS), in terms of the qualitative composition of the isolated essential oil. The isolation of the essential oil of natural products by incubate sample for 5 min at 40 ºC is described. The volatile components were identified by percentage of total peak areas comparing their retention times of GC chromatograph with NIST mass spectral library. The results show no significant difference in the seven chromatograms of perfumed powder (Pang-Rum) both with binder and without binder. Further identification was done by GC-MS. Some components of Pang-Rum with/without binder were changed by temperature and time.

***Keywords*—**GC-MS analysis, essential oils, stability, Pang-Rum.

## I. Introduction

IN ancient Thai society, the use of incense is traditional or ceremonial; it has been part of Thai society for a long time. There are many kinds of fragrances such as scented water, perfumed water, perfumed powder, marl powder, etc., but the most commonly used were perfumed water (Nam-Ob) and perfumed powder (Pang-Rum). Natural Thai perfume were divided two type; animal [1] or plant-based. Those oils derived from animals include four types of substance suitable for use in perfumes such as ambergris, castoreum, civet, and musk, while from plants are used the flowers, seed, bark, resin, and gum of plants [2].

Pang-Rum is a Thai perfumed powder used as a natural cosmetic since ancient times for the face and skin. It is made from different parts of plants and various plants. Pang-Rum was composed of white clay filler and floral water. Floral water was prepared from aromatic plants such as *Aquilaria crasna* Pierre, *Alyxia nitens* Kerr, and *Pandanus amaryllifolius Roxb.* [3]. Both the white clay filler and floral water is made by smoked incense; is composed of Frankincense, peel of Bergamot, white sugar and brown sugar. Normally, the physical and chemical stability of the fragrance can change the compounds when stored over a long period of time, by light and by temperature.

The aim of this research was the study of the aromatic chemical compounds in Pang-Rum and to compare it with Pang-Rum with binder for stability in different temperatures and for a period of zero days, one month, three months, and six months.

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## II. Materials and Methods

### A. Preparation of Samples [4]

Samples were prepared into two formulations. One was Pang-Rum (F1) without binder and another one was Pang-Rum with 10%w/w binder solution (binder is corn starch) (F2). The samples were then weighed into silicone molds of about 10 grams per specimen in each 2.5 x 2.5 $cm^2$ mold. Finally, the samples were dried by free evaporation. The samples were then sealed and stored until analysis. The preparation process is described in Fig. 1.

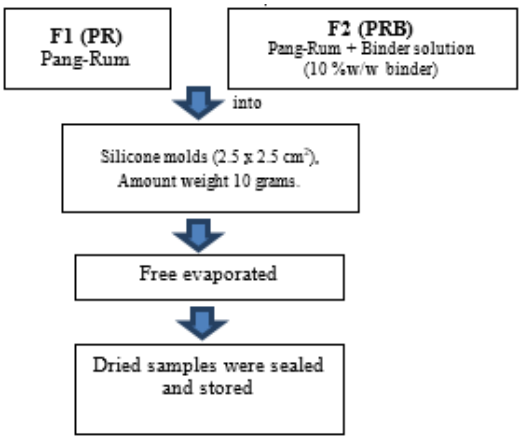


Fig. 1 Process of preparation of the sample

### B. Stability of Essential Oil

All samples were kept in a sealed package. The samples were stored at different temperatures (room temperature and 45°C) for one, three, and six months. The samples were analyzed at zero days, and one, three, and six months by GC-MS.

### C. Essential Oil Analysis

For testing, a one gram sample was analyzed using a GC sampler 80, 7890A GC system, and 5975C inert XL MSD with a Triple-Axis detector, all made by Agilent Technologies Ltd.

GC-MS conditions [5], [6]

- Column: 30 m x 250 µm id x 0.25 µm HP-5MS
- Injection: Spitless, 250 ºC, 25 µL injection volume
- Carrier: Helium (approximately 65 kPa) (9.43 psi),

constant pressure

- Oven program: 40 to 240 ºC at 20 ºC/min (30 min analysis times)
- Detection: MS in scan mode (40-400 amu); solvent delay: 3 min: transfer line: 280 ºC

*D.Identification of Volatile Components*

The identification of volatile compounds was performed by their mass spectra with data from Adam, the US National Institute of Standards and Technology (NIST) mass spectra library.

*E. Statistical Analysis*

Data obtained were analyzed using the GraphPad Prism 5 version 5.01 (GraphPad Software Inc. La Jolla, CA. USA). All values are expressed as mean±standard deviation for three replicates. Data were analyzed by ANOVA and the statistical significance differences were analyzed using the paired t-test; $p<0.05$ was considered statistically significant.

## III. Results and Discussion

The volatile components of Pang-Rum were identified by the retention times of the GC-MS analysis. The six peak area components of the oil and percentage of each constituent were summarized and presented in Table I.

Analysis of the isolate by GC-MS resulted in the identification of six components comprising 100% of total volatile components. The chromatogram of Pang-Rum was detected by mass spectrum in scan mode (40-400 amu). For six peak the presence of Camphene (27.6%), D-Limonene (40.5%), Phenylethyl Alcohol (88.4%), endo-Borneol (39.5%), 6-Octen-1-ol,3,7-dimethyl-,(R)- (38%), and Bicyclo[3.1.1]heptane,6,6-dimethyl-2-methylene-,(1S)- (23.4%), were also detected, as shown in Table II.

The chromatogram of Pang-Rum with 10%w/w binder was detected by mass spectrum in scan mode (40-400 amu). The compounds of Pang-Rum with 10%w/w binder by GC-MS were compared with the NIST library and shown in Table III.

The stabilities of the essential oils from Pang-Rum and Pang-Rum with 10%w/w binder were evaluated by GC-MS. The compounds found in Pang-Rum stored at room temperature for six months were identified as: Phenylethyl Alcohol (57.6%), Camphene (13.2%), endo-Borneol (17.6%), Citronellol (13.8%), and Bicyclo[3.1.1]heptane,6,6-dimethyl-2-methylene-,(1S)- (17.5%), as shown in Table IV.

Pang-Rum with 10%w/w binder was stored at room temperature for six months. The compounds identified, shown in Table V include, Phenylethyl Alcohol (85.6%), Camphene (13.4%), endo-Borneol (27.7%), Citronellol (21.3%), and Bicyclo[3.1.1]heptane,6,6-dimethyl-2-methylene-,(1S)- (21.4%).

The analysis of Pang-Rum stored at 45°C for six months, five peaks of compounds were identified as: Phenylethyl Alcohol (61.5%), Camphene (14.7%), Bicyclo[2.2.1]heptan-2-ol,1,7,7-trimethyl-,formate,endo-(16.8%), Citronellol (21.3%), and Bicyclo[3.1.1] heptane,6,6-dimethyl-2-methylene-,(1S)- (14.7%), as shown in Table VI.

Table VII shows the components identified of Pang-Rum with 10%w/w binder at 45°C for six months, five peaks of compounds were identified as; Phenylethyl Alcohol (62.4%) Camphene (14.2%), Bicyclo[2.2.1]heptan-2-ol,1,7,7-trimethyl-, formate, endo- (15.6%), Citronellol (13.2%), and Bicyclo[3.1.1]heptane, 6,6- dimethyl-2-methylene,(1S)- (21.4%).

TABLE I
AVERAGE OF MAIN RETENTION TIME AND TOTAL AREA PEAK OF ESSENTIAL OILS FROM PANG-RUM WITH/WITHOUT BINDER

| Formula | RT | % of area peak | | | | % Total of area peak |
|---|---|---|---|---|---|---|
| | | 0 | 1 | 3 | 6 | |
| Pang-Rum | 7.553 | 3.26 | 2.07 | 1.16 | 1.09 | 97.59 |
| | 10.236 | 16.53 | 11.66 | 15.78 | 5.64 | |
| | 10.968 | 26.66 | 20.30 | 20.95 | 27.29 | |
| | 11.112 | 41.83 | 49.64 | 50.21 | 55.47 | |
| | 11.917 | 6.00 | 9.60 | 8.05 | 7.19 | |
| | 12.302 | 2.49 | 5.74 | 3.85 | 2.87 | |
| Pang-Rum with binder | 7.553 | 2.09 | 1.69 | 0 | 4.31 | 97.50 |
| | 10.236 | 17.74 | 20.63 | 12.88 | 13.18 | |
| | 10.968 | 20.86 | 16.81 | 21.17 | 26.71 | |
| | 11.112 | 45.34 | 43.63 | 50.24 | 46.86 | |
| | 11.917 | 7.46 | 10.17 | 10.19 | 4.85 | |
| | 12.302 | 4.13 | 4.69 | 4.26 | 2.14 | |

RT=retention time

TABLE II
COMPOUNDS OF PANG-RUM BY GC-MS COMPARED WITH NIST LIBRARY

| Peak | RT | Compound | MF | MW | %Prop |
|---|---|---|---|---|---|
| 1 | 7.543 | Camphene | $C_{10}H_{16}$ | 136 | 27.6 |
| 2 | 8.870 | D-Limonene | $C_{10}H_{16}$ | 136 | 40.5 |
| 3 | 10.253 | Phenylethyl Alcohol | $C_8H_{10}O$ | 122 | 88.4 |
| 4 | 11.120 | endo-Borneol | $C_{10}H_{18}O$ | 154 | 39.5 |
| 5 | 11.922 | 6-Octen-1-ol, 3,7-dimethyl-, (R)- | $C_{10}H_{20}O$ | 156 | 38.0 |
| 6 | 12.308 | Bicyclo[3.1.1] heptane, 6,6-dimethyl-2-methylene-, (1S)- | $C_{10}H_{16}$ | 136 | 23.4 |

RT=retention time, MF=molecular formula, MW=molecular weight, %Prop= property percentage

TABLE III
COMPOUNDS OF PANG-RUM WITH 10%W/W BINDER BY GC-MS COMPARED WITH NIST LIBRARY

| Peak | RT | Compound | MF | MW | %Prop |
|---|---|---|---|---|---|
| 1 | 7.526 | Camphene | $C_{10}H_{16}$ | 136 | 29.6 |
| 2 | 8.879 | D-Limonene | $C_{10}H_{16}$ | 136 | 25.2 |
| 3 | 10.253 | Phenylethyl Alcohol | $C_8H_{10}O$ | 122 | 88.4 |
| 4 | 10981 | Isobornyl formulate | $C_{11}H_{18}O_2$ | 182 | 19.2 |
| 5 | 11.125 | endo-Borneol | $C_{10}H_{18}O$ | 154 | 39.3 |
| 6 | 11.927 | 6-Octen-1-ol, 3,7-dimethyl-, (R)- | $C_{10}H_{20}O$ | 156 | 38.7 |
| 7 | 12.308 | Bicyclo[3.1.1]heptane, 6,6-dimethyl-2-methylene-, (1S)- | $C_{10}H_{16}$ | 136 | 23.7 |

RT=retention time, MF=molecular formula, MW=molecular weight, %Prop= property percentage

TABLE IV
COMPOUNDS OF PANG-RUM STORED AT ROOM TEMPERATURE FOR 6 MONTHS

| Peak | RT | Compound | MF | MW | %Prop |
|---|---|---|---|---|---|
| 1 | 10.236 | Phenylethyl Alcohol | $C_{10}H_{16}$ | 122 | 57.6 |
| 2 | 10.968 | Camphene | $C_8H_{10}O$ | 136 | 13.2 |
| 3 | 11.116 | endo-Borneol | $C_{10}H_{18}O$ | 154 | 17.6 |
| 4 | 11.918 | Citronellol | $C_{10}H_{20}O$ | 156 | 13.8 |
| 5 | 12.299 | Bicyclo[3.1.1]heptane,6,6-dimethyl-2-methylene-, (1S)- | $C_{10}H_{16}$ | 136 | 17.5 |

RT=retention time, MF=molecular formula, MW=molecular weight, %Prop= property percentage

TABLE V
COMPOUNDS OF PANG-RUM WITH 10%W/W BINDER STORED AT ROOM TEMPERATURE FOR 6 MONTHS

| Peak | RT | Compound | MF | MW | %Prop |
|---|---|---|---|---|---|
| 1 | 10.231 | Phenylethyl Alcohol | $C_{10}H_{16}$ | 122 | 85.6 |
| 2 | 10.968 | Camphene | $C_8H_{10}O$ | 136 | 13.4 |
| 3 | 11.109 | endo-Borneol | $C_{10}H_{18}O$ | 154 | 27.7 |
| 4 | 11.914 | Citronellol | $C_{10}H_{20}O$ | 156 | 21.3 |
| 5 | 12.299 | Bicyclo[3.1.1]heptane,6,6-dimethyl-2-methylene-, (1S)- | $C_{10}H_{16}$ | 136 | 21.4 |

RT=retention time, MF=molecular formula, MW=molecular weight, %Prop= property percentage

TABLE VI
COMPOUNDS OF PANG-RUM STORED AT 45°C FOR 6 MONTHS

| Peak | RT | Compound | MF | MW | %Prop |
|---|---|---|---|---|---|
| 1 | 10.236 | Phenylethyl Alcohol | $C_{10}H_{16}$ | 122 | 61.5 |
| 2 | 10.968 | Camphene | $C_8H_{10}O$ | 136 | 14.7 |
| 3 | 11.116 | Bicyclo[2.2.1]heptan-2-ol, 1,7,7-trimethyl-, formate, endo | $C_{10}H_{18}O_2$ | 182 | 16.8 |
| 4 | 11.913 | Citronellol | $C_{10}H_{20}O$ | 156 | 12.3 |
| 5 | 12.299 | Bicyclo[3.1.1]heptane,6,6-dimethyl-2-methylene-, (1S)- | $C_{10}H_{16}$ | 136 | 14.7 |

RT=retention time, MF=molecular formula, MW=molecular weight, %Prop= property percentage

TABLE VII
COMPOUNDS OF PANG-RUM WITH 10%W/W BINDER STORED AT 45°C FOR 6 MONTH

| Peak | RT | Compound | MF | MW | %Prop |
|---|---|---|---|---|---|
| 1 | 10.236 | Phenylethyl Alcohol | $C_{10}H_{16}$ | 122 | 62.4 |
| 2 | 10.964 | Camphene | $C_8H_{10}O$ | 136 | 14.2 |
| 3 | 11.111 | Bicyclo[2.2.1]heptan-2-ol, 1,7,7-trimethyl-, formate, endo | $C_{10}H_{18}O_2$ | 182 | 15.6 |
| 4 | 11.918 | Citronellol | $C_{10}H_{20}O$ | 156 | 13.2 |
| 5 | 12.304 | Bicyclo[3.1.1]heptane,6,6-dimethyl-2-methylene-, (1S)- | $C_{10}H_{16}$ | 136 | 21.4 |

RT=retention time, MF=molecular formula, MW=molecular weight, %Prop= property percentage

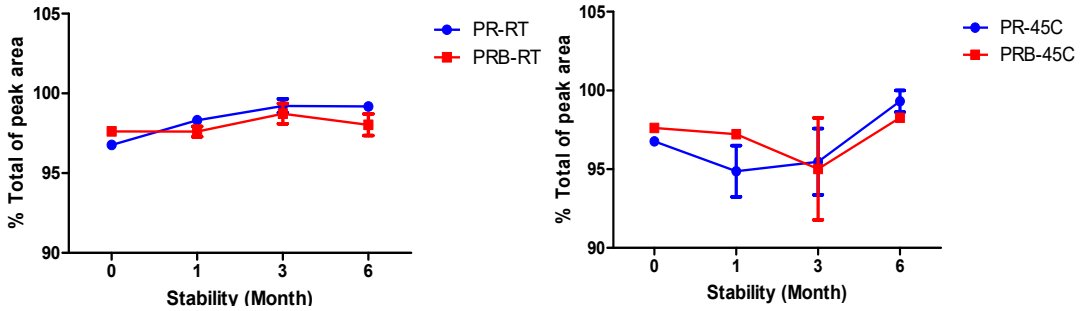


Fig. 2 Stability of essential oils from Pang-Rum with/without binder (at least 6 months)

Fig. 2 shows the percentage of total peak area of Pang-Rum with/without binder stored at room temperature and 45ºC for six months. The results found that the stability of essential oil from Pang-Rum with/without binder were not significant. Pang-Rum and Pang-Rum with 10%w/w binder presented similar major compounds from seven chromatograms. For the six month samples, the components of Pang-Rum and Pang-Rum with 10%w/w binder showed change. Meanwhile, the compounds of Pang-Rum and Pang-Rum with 10%w/w binder were similar in all conditions (room temperature and 45°C). But some components of Pang-Rum and compounds of Pang-Rum with 10%w/w binder were found to be different in the comparison of the room temperature and 45°C samples.

Both of the different Pang-Ram mass spectrum (Pang-Ram and Pang-Rum with 10%w/w binder) showed isomer compounds as 6-Octen-l-ol,3,7- dimethyl-,(R)- and Citronellol when compared with day zero (Fig. 3).

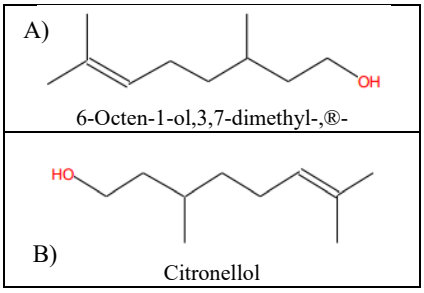


Fig. 3 Components of Pang-Rum with/without binder are isomer; A) components of Pang-Rum with/without binder for 0 day, B) components of Pang-Rum with/without binder for 6 months

## IV. CONCLUSION

In conclusion, the results show that the essential oil content and quality of Pang-Rum without binder and Pang-Rum with binder were similar all conditions. Following from [7], future

research will study the essential oil content and quality of essential oil at 4 °C and -20 °C.

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## CONFLICT OF INTEREST

The authors declare no conflict of interest with this study.

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