Encapsulation of *Michelia alba* D.C. Extract Using Spray Drying and Freeze Drying and Application on Thai Dessert from Rice Flour

Rajnibhas Samakradhamrongthai[1], Prodepran Thakeow[1], Phikunthong Kopermsub[1], and Niramon Utama-ang[2]
[1]Division of Product Development Technology, Faculty of Agro-Industry, Chiang Mai University, Chiang Mai, Thailand
[2]Lanna Rice Research Center, Chiang Mai University, Chiang Mai, Thailand
Email: blurberries@hotmail.com, {prodepran.t, niramon.u}@cmu.ac.th, phikunthong.kopermsub@nstda.or.th

***Abstract*—The objectives of this research were to investigate and characterize encapsulated powder of *Michelia alba* D.C. (MAD) extract from spray drying and freeze drying with Octenyl Succinic Anhydride starch (OSA). The results showed that moisture content, water activity and solubility of spray drying were lower than freeze drying whereas the encapsulation efficiency was higher. The X-ray diffraction results also revealed that the freeze-dried encapsulated powder exhibited less amorphous form and higher crystallinity than the spray-dried encapsulated powder. It was indicated that the encapsulated powder from spray drying and freeze drying created complexes of the OSA starch and the MAD extract which indicated high stability of encapsulated powder. In addition, the decreasing of aroma content from the MAD encapsulated powder also that volatile compounds are restored better by microencapsulation. The spray-dried encapsulated powder was taken to create the multi-core encapsulated powder and applied on Nam Dok Mai (NDM) Thai dessert. It was varied at 0, 0.5, 1, 3, and 5% w/w. The color and texture profile were analyzed and also sensory evaluation. The results revealed that NDM dessert with 1% w/w of multi-core encapsulated MAD flavor powder showed the most preferable of sensory preference. In conclusion, the spray drying was suitable for encapsulating the MAD extract. The multi-core encapsulated MAD powder at 1% w/w was the most suitable amount to apply on NDM dessert.**

***Index Terms*—*Michelia alba* D.C., encapsulation, spray drying, freeze drying, octenyl succinic anhydride starch, Thai dessert**

I. Introduction

Michelia alba D.C. (MAD) is originated from southern Asia which it has acclimatized to many regions of the world [1]. The white elongated bell-shaped flowers have a strong sweet fragrance. They are used as religious offerings or in garlands and their sweet, pungent, alluring fragrance make them ideal ingredients for aromatherapy products [2]. Flavor and aroma stability has become increasingly of interest because of the relationship between quality and acceptability of foods [3].

The properties of the microcapsules obtained from spray drying and freeze drying techniques can be influenced by emulsion or suspension properties [4]. Normally, spray-dried aromas are water-soluble, which could or could not be desirable. Freeze drying is recognized as the best method for producing high-quality dried food products, although it is less attractive than spray drying due to high energy consumption and long processing time [5].

Interestingly, the development of Thai dessert was rarely approached. Most of Thai dessert was consisted of rice flour. This component was one of the ingredients that can be affected toward dessert texture and its aroma/flavor release from both during processing time and consuming in mouth. Hence, Thai dessert considered and developed using encapsulated flavor powder, since its ingredients are mostly similar and its unique flavor/aroma will be dominance which came from Thai herbs and flowers. Variation in ingredients concentration or characteristics can modify the rheological and sensory properties of semisolid dessert, influencing consumer response [6].

This research aimed to compare the properties and characteristics of spray-dried and freeze-dried MAD encapsulated powders. The Multi-core Encapsulated MAD Flavor Powder (MEFP) was prepared from the suitable drying process to apply on NDM Thai dessert. The results provide information regarding the properties of encapsulated powder, which identify the suitable process of encapsulation on MAD extract. The application of MEFP on NDM dessert can be shown the suitable amount of MEFP that provided the most preferable sensory acceptance toward consumer and suitable textural profile of NDM dessert.

II. Materials and Method

A. Materials

The OSA starch was purchased from National Starch & Chemical (Thailand), Co., Ltd. (Bangkok, Thailand).

Manuscript received September 24, 2015; revised November 26, 2015.

doi: 10.18178/ijfe.1.2.77-85

The MAD flower was purchased from flower orchard (Nakorn Pathom, Thailand). Those were collected at 5-8 am during June 2013. The 95% ethanol was purchased from Union Science Co., Ltd. (Chiang Mai, Thailand). The NDM dessert ingredients consisted of rice flour, tapioca flour, pure refined sugar, and egg yellow color (Best Odour Brand, Best Odour Co., Ltd., Bangkok, Thailand) were purchased from Yok intertrade Co, Ltd. (Chiang Mai, Thailand). Gelatin and gum arabic were purchased from Union Science Co., Ltd. (Chiang Mai, Thailand).

B. Microencapsulation of MAD Extract Using Spray Drying and Freeze Drying

The procedure for the preparation of the emulsion was modified from Ades *et al.* [7]. The aqueous phase was prepared by dissolving the OSA starch at a suitable selected concentration (100% w/v) in deionized water at 50 ℃ while stirring for 30 min until the solution temperature reached 90 ℃. The MAD extract (10% w/v), was added into solution and stirred vigorously [8]. The emulsion was dehydrated using spay drying and freeze drying. The spray dryer (March Cool Industry Co., Ltd., Bangkok, Thailand) was operated at an inlet temperature of 150 ℃ and an outlet temperature of 50 ℃ with blower speed at 50rpm. For freeze drying process, the emulsion was placed in freezing trays and froze at −20 ℃ immediately after preparation. After 24 hours, the frozen emulsion was dried for more than 48 hours at −45°C under a pressure of less than 0.120 mbar using a freeze dryer (Model 7948030, Labconco, USA). The spray-dried and freeze-dried samples were kept at −18 ℃ for further analysis.

1) Physical properties

The encapsulated powder from spray drying and freeze drying were analyzed for yield recovery, moisture content, water activity, color value (L*, a*, b*). The moisture content calculated followed the method from AOAC NO. 934.01. The water activity was analyzed using AquaLab LITE (DECAGON Devices Inc., USA). The color value was analyzed using Hunter LAB (Colorquest XE, Hunter Lab, USA). The solubility of the encapsulated powder was examined according to the method described in Fernandes *et al.* [9].

2) Encapsulation efficiency (%EE)

The quantities of the surface content and the total content of the MAD extract were determined and calculated for %EE. Five grams of sample was washed with 70% v/v ethanol for 5 min. The total content was determined from the cleansed solvent from the encapsulated powder with 70% (v/v) ethanol for 15 min. The quantities were reported as the mean and the standard deviation of triplicate measurements [7], [9].

3) Characterization of volatile compounds

The volatile compounds were analyzed using Gas Chromatography Flame Ionization Detector (GC-FID). The volatile compounds were identified with the headspace of each sample, using Solid Phase Microextraction technique (SPME). The 85Pm Carboxen™/Polydimethylsiloxane StableFlex™ type fiber (Car/PDMS, Supelco, USA) was used. The Car/PDMS fiber was exposed for 60 sec in the headspace of a septum-capped vial containing 2g of the samples. Subsequently, the fiber was directly injected into the injection port of a gas chromatograph (GC-2010, 05853, Shimadzu, Japan). The FID was operated with the sampling rate at 40 msec, an air flow rate of 400mL min^{-1}, and with the source temperature at 230 ℃. The GC was operated on the DB-WAX column (30m×0.53mm, i.d., 1.50μm film thickness) (Model 125-7333, Agilent Technologies, Inc., USA), and helium was used as the carrier gas at a flow rate of 50.0mL/min. The temperature program was started with an initial temperature of 40 ℃, which was then heated up to 250 ℃ at the rate of 7 ℃/min and held for 5 min at 250 ℃. The MS was operated in the electron impact mode with electron energy of 70eV and with the scan over range of 20-300 amu, the source temperature being 230 ℃. The obtained mass spectra were preliminarily interpreted by comparing with those of the enhanced chemstation version D00.00.38 (Agilent Technologies), the mass spectral search library of the National Institute of Standards and Technology (NIST, Gaithersburg, USA).

4) Characterization of encapsulated powder

- Morphology of encapsulated powder: The microstructures obtained from both spray drying and freeze drying were examined using a scanning electron microscope (SEM, JSM5410-LV, JEOL, Japan). Photographs were taken at an excitation voltage of 10kV [10].
- Glass transition temperature (T_g): the encapsulated samples were weighed (5±0.2mg) in an aluminum pan and sealed. The measurement was conducted by differential scanning calorimeter (Diamond DSC, Perkin Elmer, Inc., OH, USA), using a liquid nitrogen cooling system (Intracool 2P, TA instruments, NC, USA). The operating conditions were as follows: nitrogen flow rate at 20mL/min and at temperatures ranging from 20 ℃ to 120 ℃ at the rate of 10 ℃ per min. [4].
- X-Ray Diffraction (XRD): The formation of the encapsulated powder was verified using XRD (Miniflex II, Rigaku Corporation, Japan) providing the Cu Kα1 bean radiation (λ=0.154nm). The diffractograms were obtained under the condition of 40kV and 30mA, with the scanning angle 2θ set from 5° to 30° with a scanning rate of 0.02 °/sec [11].

C. Preparation of Multi-Core Encapsulated MAD Flavor Powder

The suitable encapsulation process was selected from *B part* to prepare for MEFP. The mixtures were prepared according to a method described by Alvim and Grosso [12]; Butstraen and Salaün [13] with modifications. The aqueous phase was prepared by dissolving gelatin and gum arabic separately in deionized water at 50 ℃ while stirring for 30 min until the solution dissolved into homogenous mixture. Pandan aroma was infused into gelatin mixture. The MAD flavor powder from spray

drying was dispersed into gelatin mixture at 2.5% w/w under magnetic stirring condition (1000rpm). The solution of gum arabic was added together to create Gelatin-Gum Arabic system (GGA). The pH of GGA mixture was adjusted to 4.0±0.2 using 10% v/v acetic acid and then slowly cooled to 0℃ to create complexes of multi-core complexes in GGA system. The mixture was stirred for another 15 min for creating multi-core complexes extensively under magnetic stirring condition (500rpm). The precipitated microspheres were cleansed twice by decanting with distilled water and collected by centrifugation at 5000g for 5 min. (universal 320R, Zentrifugen, Tuttlingen, Germany). The microspheres were dehydrated using freeze dryer. The microcapsules obtained from freeze drying were directly weighed and stored in desiccators for further experiment.

D. Application of Multi-Core Encapsulated Powder on NDM Dessert

The NDM dessert was adjusted and modified from basic formulation from Jueyjareon. [14]. The fundamental formulation in this experiment was adjust and added tapioca starch to improve its texture which consisted of rice flour (18.0%), tapioca flour (2.0%), sugar (23.0%), and water (57%). The experiment was designed using Completely Randomized Design (CRD). The variation of multi-core encapsulated MAD flavor powder was varied from 0.5%, 1%, 3%, and 5% w/w with 0% w/w of flavor powder as a control [8]. The production of this dessert was prepared in steam boiler which must preheated temperature toward 98±2℃. The rice flour and tapioca starch were mixed together separately from sugar that was melt in water until it was a homogeneous. The syrup from sugar and water was poured into mixture of rice flour and tapioca starch then knead until it becomes homogeneous. The mixture was poured into a preheated cup (98±2℃) inside a steam boiler with amount of 12.5g each and then closed the steam boiler lid for 15 min. The finished NDM dessert was taken to measure for color value (L*, a*, b*) and texture profile.

1) Color measurement

The color was analyzed using Hunter LAB (Colorquest XE, Hunter Lab, USA). The light source was Illuminant D65 and the Observation was 10 degree. The color value were reported in CIELab color system where L* (Lightness), a*(negative value means green and positive value means red), and b*(negative value means blue and positive value means yellow) were expressed. All samples were conducted in triplication.

2) Texture Profile Analysis (TPA)

TPA of NDM desert was performed on a TA-XT plus texture analyzer (TA-XT plus, Stable Microsystems, Surrey, UK) using an aluminum probe P/45C (diameter 45mm). The TPA method was conducted under condition as the following: pre-test speed, 2mm/sec; post-test speed, 1mm/sec; rupture test distance, 1%; measurement distance, 40% deformation; force, 0.10kg; and auto trigger force, 0.020kg. The distance from the platform was 30.0mm, with double compression performed in intervals of 10 sec between two compressions [15]. The textural parameters calculated were hardness (g.Force), adhesiveness (g.Force), cohesiveness (dimensionless), springiness (cm), gumminess (g.Force), and chewiness (g.Force). Ten measurements were performed on each sample to obtained mean measurement for that sample at room temperature.

3) Determination of aroma release profile of NDM dessert

- Determination of Pandan aroma from outer shell

The NDM dessert was analyzed for Pandan aroma release profile during steam process. Two mL of dessert slurry mixture was added into simulated steam condition in capped 20mL vial. Heated water bath was preceded as the production of the production of NMD production with controlled temperature of 98±2℃. Evaporated volatile compound from static head space was sampling at 0, 5, 7.5, 12.5, and 15 min which the considering time for NDM to be finished was 15 min [16].

- Determination of MAD aroma from inner
- Encapsulated Powder in Simulated Artificial Saliva

The MAD aroma release of the finished NDM dessert was analyzed in simulated artificial saliva (SSF) [7]. The Chopped NDM dessert (20mg) was incubated in SSF; the pH was adjusted to 7.2 by potassium hydroxide. The α-amylase activity used was 100 unit/mL, as the average activity found while chewing [7]. The incubation was set in a 2mL glass vial sealed by a screw cap covered with an aluminum foil filled with 2mL SSF at 37℃ under continues stirring (12rpm). Evaporated volatile compound from static head space was sampling 0, 1, 2, 3, 4, and 5 min. which considered time for maximum release in oral cavity of NDM dessert.

- Gas Chromatograph Flame Ionization Detector analysis (GC-FID)

The extent of aroma released following the incubation in simulated conditions was measured by extracting aroma from the reaction medium and quantification by GC-FID. Gas chromatograph analysis was performed the condition as previously described. Standards calibration curve of aroma were in order to calculate amount of each aroma from NDM dessert.

4) Sensory acceptance test

All variation formula of NDM dessert was presented in disposable closed lid plastic cup coded with three-digit number. Tests were performed in individual air-conditioned booths (25℃) in the Sensory Evaluation and Consumer testing Laboratory (Division of product development technology, Faculty of Agro-Industry, Chiang Mai University, Chiang Mai, Thailand). The NDM dessert was evaluated by untrained consumers (n=50) using as 9-point hedonic scale [17] with NDM dessert attributes (appearance, color, aroma, flavor, taste, texture, overall liking) [14]. The tested samples were also evaluated for product acceptance. The most preferable formulation in sensory attributes with high product acceptance was selected to be the most suitable formulation for applying the multi-core encapsulated MAD flavor powder on NDM dessert.

E. Statistical Analysis

All the data were collected in triplicate. Analysis of Variance (ANOVA) was performed and using the Duncan's Multiple Range Test (DMRT) for mean separation. The data regarding the samples from both spray drying and freeze drying were presented as mean values ±standard deviations and analyzed using the t-test statistical analysis. The analysis of all data was conducted using SPSS 17.0 (SPSS Inc., IBM Corp., Chicago, IL, USA), with the significant level determined at 95% confidence limit (p<0.05).

III. Result and Discussion

A. Comparison of Spray Drying and Freeze Drying Powder

The maximum OSA starch (100% w/v) was used in this experiment to screen for the most suitable process of encapsulation of MAD extract. The yield recovery from freeze drying was higher than that from spray drying at 50.01% and 22.42% respectively, (Table I), which conformed to the finding of Santo *et al.* [18] who compared spray drying and freeze drying to obtain the powdered *Rubrivivax gelatinosus* biomass. The findings showed that spray drying registered lower in moisture content, water activity, and mass recovery, whereas there were significant results for yield recovery.

TABLE I. Comparison of MAD Encapsulated Powder from Spray Drying and Freeze Drying

Properties of encapsulated powder	Spray Drying	Freeze Drying
Yield recovery (%)	22.42 ±0.55b	50.01 ±0.18a
Moisture content (%)	0.70 ±0.08b	1.28 ±0.01a
Water activity	0.610 ±0.004b	0.656 ±0.007a
Color value L*	87.93 ±0.34a	72.84 ±0.23b
Color value a*	3.35 ±0.07b	9.45 ±0.36a
Color value b*	14.38 ±0.27b	21.58 ±0.76a
Solubility (%)	81.89 ±0.70b	98.67 ±0.07a
Surface content (%)	0.54 ±0.03a	2.4 ±0.06b
Extract recovery (%)^{ns}	73.61 ±2.91	72.25 ±1.50
Encapsulation efficiency (%EE)	92.62 ±0.45a	66.74 ±1.19b
T_g ^{ns} (℃)	75.20 ±0.93	70.65 ±0.50

[1]Mean values and standard deviation. [a,b]Letters in the same row with different superscripts mean differ significantly (p<0.05) by the t-test

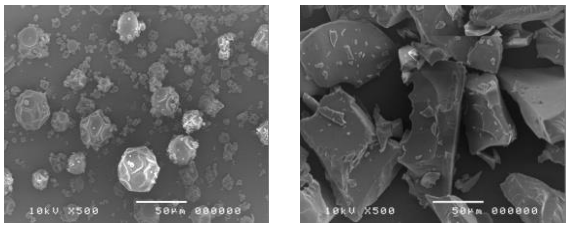
The lower yield of spray drying resulted from the adhesiveness of the slurry solid, and it has been responsible for the lower yield recovery in the case of spray drying which caused the product to adhere to the spray dryer chamber, leading to losses during the process. However, the moisture content, water activity, and solubility of spray drying were lower than freeze drying, which indicated the high stability of the encapsulated powder. These parameters pointed to the fact that the spray drying method was more effective in removing water which can be explained by the extreme contact between the microcapsule particles and the hot air in the chamber. The lower moisture content registered by the spray-drying technique demonstrated that this technique was more effective than freeze drying. The spray-dried product exhibited higher color value of lightness (L*), whereas its color values a* and b* were lower. These results demonstrate that the spray-dried powder was lighter, less red, and less yellow, which means that it had more saturated color as compared to the freeze-dried powder. These differences were due to the higher dynamics of the spray-drying process, in which it was temperature and pressure that changed the color of the final product. In addition, the color attributes suggested that the spray-dried powder provided higher consistency of slurry prior to the encapsulation process. Additionally, another factor that explained the differences in the color value was the difference in the water content of the encapsulated powders since the drying process modifies the surface of a product, altering its reflectiveness and color [19].

The encapsulation efficiency (%EE) of spray drying was higher than that of freeze drying, and these were 92.62% and 66.74%, respectively, whereas the observed surface content of spray drying was lower than that of freeze drying, which were 2.40% and 6.82% respectively (Table I). The disparity in the values of the encapsulation efficiency is indicative of the higher retention of volatile compounds during drying. This indicated that spray drying can create microcapsules in the film-forming shell at the last stage of drying, resulting in high incidence of entrapment of extract content which leads to low content of surface content [20]. On the other hand, freeze drying promotes droplet-to-droplet interaction in the emulsion until the drying stage, which causes freeze drying to consume more time than spray drying, resulting in inconsistency in the entrapment of the extract of the freeze-dried encapsulated powder which leads to low incidence of entrapment of extract content and thereby, high surface content [4]. These results suggested that spray drying has higher stability in encapsulation than freeze drying.

B. Characterization of Encapsulated Powder

1) Morphology of encapsulated powder

The microstructures of the encapsulated powder from spray drying and freeze drying were different (Fig. 1). The spray-dried powder showed a spherically regular shape with a shallow dent of shrinkage which had happened during the early stage of drying and cooling. These results are conformed to the findings of the investigation of Sahin-Nadeem *et al.* [21]. In contrast, the freeze-dried powder was more likely to develop an irregular crystalline-like shape with sharper edges, broken glass-like surface, and brittle texture due to the lyophilized process [4]. The asymmetrical shape is usually the result of homogenization of the core material in the matrix solution, which creates an irregular crystalline-like shape before lyophillization [22]. The sample is frozen at temperatures between −90°C and −40°C and then dried by direct sublimation under low pressure and reduced temperature (between −90°C and −20°C). After drying, the brittle matrix obtained can be broken or ground into smaller pieces [22].



(a) (b)

Figure 1. SEM photographs of microencapsulation of MAD flower extract using (a) spray drying and (b) freeze drying.

2) Glass transition temperatures (T_g) of encapsulated powders

The glass transition temperatures of the encapsulated powders from spray drying and freeze drying were 75.2 ℃ and 70.65 ℃, respectively. The results was conformed to the result obtained by Chen *et al.* [4] that the T_g of the spray-dried powder was lower than that of the freeze-dried powder, and this provided higher stability under the storage conditions. The values of T_g obtained from the analyses of the encapsulated powders from spray drying and freeze drying were above 70 ℃, which indicates that the material transformed to glass state when the temperature of the sample goes beyond 70 ℃ and that it can be stored at a temperature below 70 ℃ at 25% ambient relative humidity, which has an inclination toward the finding by Desobry *et al.* [23] in an investigation of the comparison of spray drying and freeze drying for β-carotene encapsulation and preservation. These results demonstrated that encapsulated powder from spray drying provided higher stability than encapsulated powder from freeze drying and also that spray drying is a suitable process to encapsulate extract from MAD.

3) X-Ray Diffraction (XRD) of encapsulated powders

The changes in the degree of crystallinity of the samples were analyzed using XRD. The diffraction patterns were compared to known V-type polymorphs as also with the diffraction patterns of the OSA starch itself. The results of the XRD analysis showed that all the samples were in the amorphous form (Fig. 2). The non-processed OSA starch had a typical diffraction which had Bragg angles of 2θ for A-Type diffraction (11 °, 15 °, 17 °, and 18 °) and B-Type diffraction (11 °, 15 °, 17 °, and 22 °). The mixing of the diffraction angles suggests that the non-processed OSA starch used in this experiment had C-Type crystallinity diffraction (11 °, 15 °, 17 °, 18 °, 22 °, 23 °, and 24 °) which mixed between the A-Type diffraction and the B-Type diffraction. The encapsulated powders from spray drying (red color) and freeze drying (blue color) created complexes of the OSA starch and the MAD extract, resulting in V-type polymorphs which provided the Bragg angle of 2θ for the V_7-type (13 ° and 18 °) and the V_{6h}-type (20 °); also, there was the C-type crystalline pattern (11 °, 15 °, and 23 °) of the OSA starch which remained unchanged. This indicated that the OSA starch did not form complexes with the MAD extract completely. The freeze-dried encapsulated powder exhibited less amorphous form and higher crystallinity than the spray-dried encapsulated powder, which indicates that the spray-dried encapsulated powder had the stability to retain extract over the freeze-dried encapsulated powder [24].

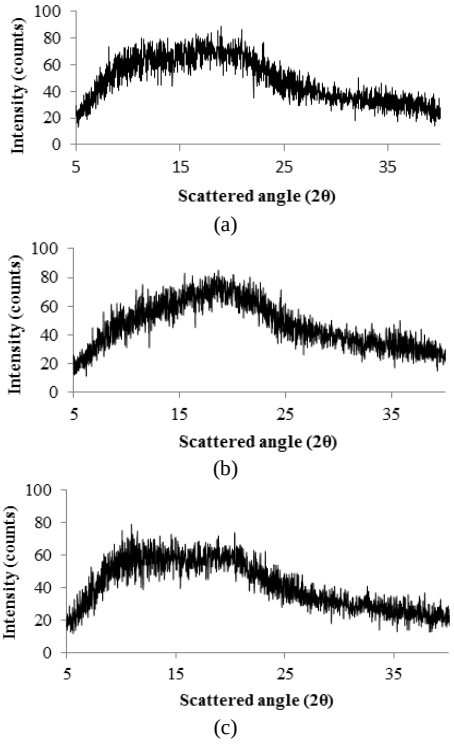


Figure 2. X-Ray diffraction scans of (a) non-process OSA starch, (b) spray-dried encapsulated powder and (c) freeze-dried encapsulated powder.

C. Comparison of Volatile Compounds from MAD Extract and Microencapsulated Powder

The volatile compounds of the MAD extract were compared to the MAD spray-dried encapsulated powder and the freeze-dried encapsulated powder. The volatile compounds detected from the MAD extract, the MAD freeze-dried powder, and the MAD spray-dried powders were quite similar (Table II). However, there were changes in the percentages of the detected volatile compounds. The MAD extract showed a high percentage of 2-methyl butanoic acid (78.69%) and lilac aldehyde (27.07%), followed by linalool (14.41%) and verbenone (7.04%), whereas in the MAD dried powders only lilac aldehyde, linalool, verbenone, and terpendiol were detected. The MAD extract showed the presence of the characteristic odors of MAD, which were 2-methyl butanoic acid and linalool [25]. Therefore, there were lower molecular weight volatile compounds that were undetectable in the MAD freeze-dried powder and the MAD spray-dried powder. Those volatile compounds were lost during the entrapment from encapsulation [26]. Interestingly, there was one significant change of an increase in an aroma compound: terpendiol was detected to increase in quantity from the extract to the encapsulation powder. The increasing amount of higher molecular weight volatile compounds happened because of the loss of low molecular weight volatile compounds, which provided more space for entrapment on the SPME fiber to get attached to since the lower molecular weight volatile compounds were lost and entrapped through the

encapsulation process. The observation of this incident was reported in Baranauskienė *et al.* [26], as well, as volatile hydrophobic compounds were considerably lost during the encapsulation and the drying process. Besides, Terta *et al.* [27] also reported that compounds with higher relative volatility possessed lower retention aroma release.

TABLE II. IDENTIFIED VOLATILE COMPOUNDS AND PEAK AREA PERCENTAGE FROM MAD POWDER AND EXTRACT

Compounds	Peak area (%)		
	MAD extract	MAD Freeze-dried powder	MAD Spray-dried powder
2-methyl butanoic acid	78.69±1.17	Not detect	Not detect
terpinolene	0.19±0.01	Not detect	Not detect
diethyl malonate	0.20±0.01	Not detect	Not detect
phenyl ethyl alcohol	15.30±0.12	0.21±0.03	Not detect
lilac aldehyde	27.07±0.05	16.91±0.01	Not detect
linalool	14.41±0.09	9.69±0.18	1.86±0.01
verbenone	7.04±0.05	6.97±0.46	1.94±0.02
terpendiol	0.91±0.01	13.24±0.05	13.24±0.35

These results explain the greater ability of low molecular weight compounds to evaporate and also to entrap inside the hydrophobic cavity created from the conformation of the polysaccharide structure [26]. Similar findings were discovered in many other studies also that there was a decrease in the quantity of volatile compounds during the drying process, as stated in the research of Yahya *et al.* [28]. The encapsulation process also reveals a decrease in the aroma from the MAD extract. All the detected aromas, except terpendiol, were observed to have decreased. The MAD encapsulated powder showed a decrease in the amount of the aroma content, which suggests that volatile compounds are restored better by microencapsulation which proved that there was good maintenance of aromatic substances inside the microcapsules. All results from this part showed that the spray drying was more suitable than freeze drying to encapsulate MAD extract. Thus, the MAD flavor powder for the multi-core encapsulated powder was created from spray drying process.

D. Application of Multi-Core Encapsulated MAD Flavor Powder on NDM Dessert

1) Color values and TPA of NDM dessert

The multi-core encapsulated flavor powder was applied in NDM dessert with variation from 0.5-5% w/w together with non-flavor powder. The results showed that Color value a* and b* of NDM dessert was differently significant in range of 0.21-0.24 and 7.01-7.03, respectively.

The TPA of NDM dessert showed significant different in all treatment; hardness profile was in range of 2714.63-4495.82 g.Force, adhesiveness profile was in range of –188.10 - –145.43 g.Force, cohesiveness profile was in range of 0.69-0.77, springiness profile was in range of 0.61-0.75cm, gumminess profile was in range of 1873.93-3482 g.Force, and chewiness profile was in range of 119.29-2481.98 g.Force as shown in Table III. The increasing multi-core MAD flavor powder affected toward texture of NDM dessert significantly. The TPA results indicated that increasing multi-core flavor powder affected to decrease hardness, cohesiveness, springiness, gumminess and chewiness. NDM dessert that contained multi-core MAD flavor powder was softer than the NDM that contained only the dessert ingredients. It suggested that hardness was decreased because of the volume of the samples were occupied by the microcapsules consisted of gelatin and gum arabic. The same results was observed for the cohesiveness, springiness, gumminess and chewiness obtained, where samples containing the microcapsules showed decreasing values of these parameters as suggested in Santos *et al.* [15] investigation. In contrast, the texture profile on adhesiveness was increased when applied more multi-core flavor powder which suggested that the sample had less adhesive and less sticky. The sample with more than 3% of multi-core flavor powder showed the adhesive to be decreased which implied that the product repossessed highly adhesive and stickier again as suggested in research of Santos *et al.* [15], both of which are unacceptable characteristics of NDM dessert.

TABLE III. COLOR VALUE AND TEXTURE PROFILE ANALYSIS OF NDM DESSERT

Flavor powder	Color value			Hardness (g.Force)	Adhesiveness (g.Force)	Cohesiveness	Springiness (cm)	Gumminess (g.Force)	Chewiness (g.Force)
	L^{*ns}	a*	b*						
0.0%	36.54±0.05	0.22±0.01c	7.01±0.03b	4426.41 ± 304.60a	-180.80 ± 29.98a	0.75±0.03a	0.75±0.08	3310.81±276.70a	2481.98 ± 230.60a
0.5%	36.59±0.04	0.21±0.01c	7.01±0.01b	4427.35 ± 281.91a	-181.54 ±3 1.85c	0.75±0.03a	0.75±0.08a	3390.29±336.90a	2506.59 ± 237.20a
1.0%	36.60±0.01	0.22±0.01bc	7.01±0.01ab	4495.82 ± 319.15a	-175.53 ± 25.91b	0.77±0.10a	0.75±0.12a	3482.02±612.93a	2621.65 ± 636.98a
3.0%	36.59±0.04	0.23±0.02ab	7.02±0.01a	3376.55 ± 269.86b	-145.43±9.38a	0.74±0.13a	0.74±0.12b	2514.72 ± 0.549.59b	1860.39 ± 485.56b
5.0%	36.60±0.03	0.24±0.02a	7.03±0.02a	2714.63 ± 261.79c	-188.10 ± 13.47d	0.69±0.09b	0.61±0.12c	1873.93±355.63c	1119.29 ± 172.83c
***p*-value**	<0.876	<0.001	<0.014	<0.001	<0.001	0.007	<0.001	<0.001	<0.001

The different letters in the same column mean significant difference ($p \leq 0.05$)

2) Determination of Pandan aroma release profile during production

The results showed that Pandan aroma was deceasing over time of production. The Pandan aroma release content was not demonstrated to be increased as expected as it was decreasing in time-course production (Fig. 3). The retention of Pandan aroma in NDM dessert production was decreased due to specific interactions with rice starch and tapioca which were able to form another complex with amylose in starch dispersions as recalled as gelatinization. The retentions have been decreased due to such complexes recreation. Those multi-core flavor powders interacted with amylopectin through a mechanism other than complexion. Previous studies have demonstrated the importance of amylopectin to aroma retention [29]. These results also explained the greater ability of gelatinization to re-entrap the release Pandan aroma from multi-core flavor powder into starch matrices of NDM dessert. Therefore, the gelatinization of NDM dessert was affected in the positive way toward the retaining of Pandan aroma.

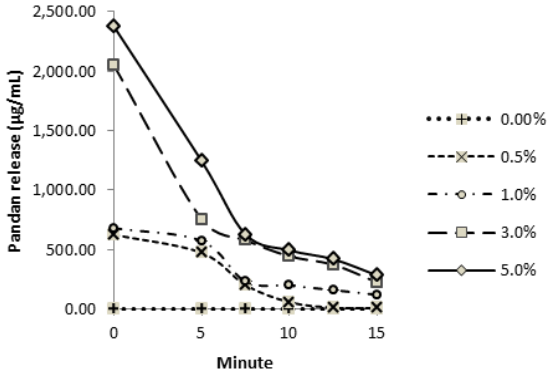


Figure 3. Pandan aroma content releases from NDM dessert production.

3) Determination of MAD flavor powder release profile in simulated artificial saliva

The finished products from section *E-(1)* were chopped in fine pieces and added into vial with simulated artificial saliva at controlled temperature water bath (37±2 ℃). The release of aroma of 2-methyl butanoic acid, linalool and verbenone was increased over time-course condition as expected (Fig. 4). This showed that multi-core encapsulated flavor powder was a success to retain aroma from MAD extract and controlled its release through enzymatic mechanism from simulated artificial saliva condition. In this experiment, release profile of main aroma compounds were release less than aroma release from only MAD flavor powder. This part showed that multi-core flavor powder was partially degraded because of enzymatic reaction also happen with starch matrix. The hydrolysis rate found to be reduced compare to uncomplex the multi-core encapsulated flavor powder as reported in researches from Ades *et al.* [7]. The findings from MAD aroma release from NDM desserts showed that the controlled release of MAD aroma from NDM dessert was release through enzymatic reaction of α-amylase. This suggested that the enzymatic reaction was involved in the release of MAD and it is can be applied to product that required releasing active ingredients in consumer oral cavity. The result showed success of retained aroma from MAD extract with controlled release property through enzymatic mechanism from SSF condition as suggested in Ades *et al.* [7] (Fig. 4). In this experiment, release profile of main aroma compounds were release less than aroma release from only MAD flavor powder. This part showed that multi-core flavor powder was partially degraded also because of enzymatic reaction.

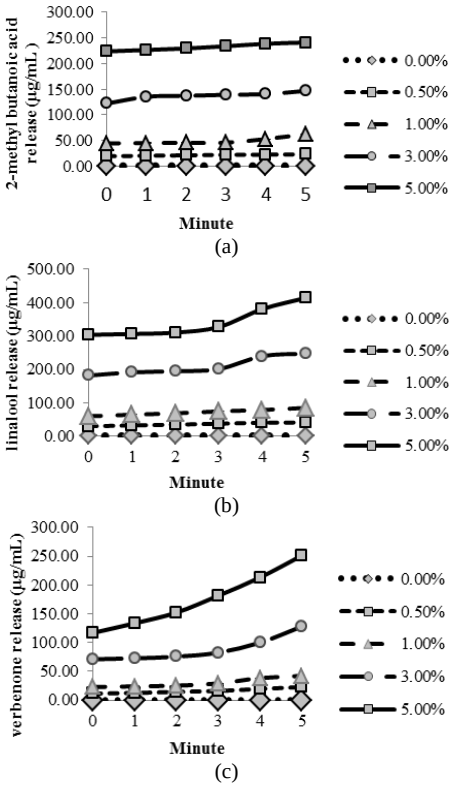


Figure 4. Release of main aroma compounds during incubation in stimulated saliva fluids (pH 7.0±0.2, 37 ℃). Aroma release was presented as the amount from static head space; (a) 2-methyl butanoic acid, (b) linalool, and (c) verbenone.

TABLE IV. SENSORY EVALUATION OF NDM DESSERT

Flavor powder	Appearance	Color	Aroma	Flavor	Taste	Texture	Overall liking	Acceptance (%)
0.0%	6.0±0.6a	6.1±0.5b	6.0±0.6b	6.1±0.7b	6.0±0.6a	5.9±0.7ab	6.0±0.7b	98.0
0.5%	6.0±0.7a	6.3±0.5a	6.0±0.6b	6.0±0.7b	6.0±0.7a	5.9±0.7ab	6.1±0.7b	98.0
1%	6.1±0.4a	6.2±0.5a	6.3±0.6a	6.3±0.7a	6.1±0.5a	6.0±0.6a	6.2±0.7a	100.0
3%	5.7±0.5b	6.2±0.5ab	6.0±0.5b	5.6±0.6c	6.0±0.6a	5.8±0.6b	5.8±0.5c	90.0
5%	5.5±0.5c	5.9±0.3c	5.7±0.5c	5.6±0.5c	5.8±0.4b	5.5±0.5c	5.6±0.5c	76.0
***p*-value**	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	

The different letters in the same column mean significant difference ($p \leq 0.05$)

4) Sensory acceptance of NDM dessert

All attributes showed sensory acceptance rating was in range of 5.5-6.3. The results showed that all attributes were significantly different when increased multi-core flavor powder up more than 1%, resulting to decrease sensory preferences lower than 6.0. The results suggested that the suitable amount of the multi-core encapsulated flavor powder for NDM dessert was 1% w/w, provided the highest rating score among all of treatments with acceptance percentage of 100% (Table IV).

IV. Conclusions

The findings of this research revealed that the microencapsulation efficiency of spray drying was higher than that of freeze drying. In addition, the moisture content, water activity, and solubility of spray drying were lower, all of which indicate higher stability. Comprehensively, it can be concluded that spray drying produced powder with superior properties and exhibited better protection toward the core materials. Moreover, the NDM dessert contained 1% w/v of MEFP showed the most preferable of sensory preference. The consumer acceptance of NDM desert with 1% of multi-core encapsulated flavor powder showed that NDM dessert provided sensory rating score in range of 6.0-6.3. This showed a success on applying the MEFP in NDM dessert with high sensory rating and high consumer acceptance.

Acknowledgement

This research was financially supported by NSTDA-University-Industry Research Collaboration (NUI-RC) under National Science and Technology Development Agent (NSTDA) and the research grants from Chiang Mai University.

References

[1] T. Smitinand, "Thai plant names," revised ed., The Forest Herbarium Royal Forest Department, Bangkok, 2001, pp. 355.
[2] N. Bunyapraphatsara, "Herbs plant folklore," Medicinal Plant Information Center, Mahidol University, Bangkok, 1996, pp. 712.
[3] P. Landy, C. Druaux, and A. Voilley, "Retention of aroma compounds by proteins in aqueous solution," *Food Chem.*, vol. 54, pp. 387-392, 1995.
[4] Q. Chen, F. Zhong, J. Wen, D. McGillivray, and S. Y. Quek, "Properties and stability of spray-dried and freeze-dried microcapsules co-encapsulated with fish oil, phytosterol esters, and limonene," *Dry Technol*., vol. 31, pp. 707-716, 2013.
[5] J. J. Schwegman, "Understanding the physical properties of freeze-dried materials," *Innov. Pharm. Technol.*, vol. 29, vol. 72-74, pp. 76-77, 2009.
[6] C. Arancibiam, C. Castro, L. Jublot, E. Costell, and S. Bayarri, "Colour, rheology, flavor release and sensory perception of dairy desserts, influence of thickener and fat content," *LWT-Food Sci. Technol.*, vol. 62, pp. 408-416, 2015.
[7] H. Ades, E. Kesselman, Y. Ungar, and E. Shimoni, "Complexation with starch for encapsulation and controlled release of menthone and menthol," *LWT-Food Sci. and Technol*., vol. 4, pp. 277-288, 2012.
[8] R. Samakradhamrongthai, "Extraction of Champaca (*Michelia champaca* L.) essential oil and its application in instant Champaca-flavored tea powder," M.S. thesis, Div. Product Development Technology, Chiang Mai Univ., Chiang Mai, Thailand, 2011.
[9] R. V. B. Fernandes, S. V. Borges, and D. A. Botrel, "Gum arabic/starch/maltodextrin/ inulin as wall material on the microencapsulation of rosemary essential oil," *Carbohyd. Polym.*, vol. 101, pp. 524-532, 2014.
[10] I. Ferreira, S. Rocha, and M. Coelho, "Encapsulation of antioxidants by spray-drying," *Chem. Eng. Trans.*, vol. 11, no. 2, pp. 713-717, 2007.
[11] R. Bhosale and R. Singhal, "Effect of octenyl succinilation on physicochemical and functional properties of waxy maize and amaranth starches," *Carbohyd. Polym.*, vol. 68, pp. 477-456, 2007.
[12] I. D. Alvim and C. R. F. Grosso, "Microparticles obtained by complex coacervation: Influence of the type of reticulation and the drying process on the release of the core material," *Ciência e Technologia de Alimentos*, vol. 30, no. 4, pp. 1069-1076, 2010.
[13] C. Butstraen and F. Salaün, "Preparation of microcapsules by complex coacervation of gum arabic and chitosan," *Carbohydr. Polym.*, vol. 99, pp. 608-616, 2014.
[14] N. Juyjaroen, "Development of Khanom Nam-Dok-Mai to set the specific of Thai standard," M.S. thesis, Dept. Agro-Industry Product Development, Kasetsart Univ., Bangkok, Thailand, 2003.
[15] M. G. Santos, *et al.*, "Coencapsulation of xylitol and mentol by double emulsion followed by complex coacervation and microcapsule application in chewing gum," *Food Res. Int.*, vol. 66, pp. 454-462, 2014.
[16] Z. Dong, *et al.*, "Morphology and release profile of microcapsules encapsulating peppermint oil by complex coacervation," *J. Food Eng.*, vol. 104, pp. 455-460, 2011.
[17] A. V. A. Resurreccion, "Consumer sensory testing for product development," An Aspen Publication, Maryland, 1998.
[18] E. F. E. Santo, L. K. F. Lima, A. P. C. Torres, G. Oliveira, and E. H. G. Ponsano, "Comparison between freeze and spray drying to obtain powder *Rubrivivax gelatinosus* biomass," *LWT - Food Sci. Technol.*, vol. 33, no. 1, pp. 47-51, 2013.
[19] P. J. Fellow, *Technologia do Processamento de Alimentos: Princ ́ios e pr áTica*, 2nd ed., Porto Alegre: Artmed, 2006, pp. 602.
[20] E. Dickinson, "Hydrocolloids at interfaces and the influence on the properties of dispersed systems," *Food Hydrocoll.*, vol. 17, pp. 25-39, 2003.
[21] H. Sahin-Nadeem, M. Torun, and F. Ozdemir, "Spray drying of the mountain tea (*Sideritis stricta*) water extract by using different hydrocolloid carriers," *LWT - Food Sci. Technol.*, vol. 44, pp. 1626-1635, 2011.
[22] N. J. Zuidum and E. Shimoni, "Overview of microencapsulated for use in food products or processes and methods," in *Encapsulation Technologies for Active Food Ingredients and Food Processing*, N. J. Zuidam and V. A. Nedovic, Eds., New York, NY, USA: Springer Science and Business Media, LLC., 2010.
[23] S. A. Desobry, F. M. Netto, and T. P. Labuza, "Comparison of spray-drying and freeze drying for β-carotene encapsulation and preservation," *LWT - J. Food Sci. Technol.*, vol. 62, pp. 1158-1162, 1997.
[24] P. Le Bail, C. Rondeau, and A, Buléon, "Structural investigation of amylose complexes with small ligands: Helical conformation, crystalline structure and thermostability," *Int. J. Biol. Macromol.*, vol. 35, pp. 1-7, 2005.
[25] W. Pensuk, T. Padumanond, and C. Pichaensoonthon, "Comparison of the chemical constituents in *Michelia alba* D.C. flower oil extracted by steam distillation, hexane extraction and enfleurage method," *J. Thai Trad. Altern. Med.*, vol. 5, no. 1, pp. 30-39, 2007.
[26] R. Baranauskienė, P. R. Venskutonis, K. Dewettinck, and R. Verhé, "Properties of oregano (*Oregaumvulgare* L.), citronella (*Cymbopogon citratus*) and marjoram (*Origanum majorana*) flavors encapsulated into milk protein-based matrices," *Food Res. Int*., vol. 39, pp. 413-425, 2006.
[27] M. Terta, G. Blekas, and A. Paraskevopoulou, "Retention of selected aroma compounds by polysaccharide solutions: A thermodynamic and kinetic approach," *Food Hydrocoll.*, vol. 20, no. 6, pp. 863-871, 2006.
[28] F. Yahya, T. Lu, R. C. D. Santos, P. J. Fryer, and S. Bakalis, "Supercritical carbon dioxide and solvent extraction of 2-acetyl-1-pyrroline from Pandan leaf: The effect of pre-treatment," *J. Supercrit. Fluids*, vol. 55, pp. 200-207, 2010.
[29] A. Seuvre, E. Philippe, S. Rochard, and A. Voilley, "Kinetic study of the release of aroma compounds in different model food systems," *Food Res. Int.*, vol. 40, pp. 480-492, 2007.

Rajnibhas Samakradhamrongthai was born on May 3, 1977 in Chiang Mai Province, Thailand. He received his M.S. degree (Agro-Industrial Product Development) from Chiang Mai University, Thailand in 2011. His major field of study is Product Development and Technology. He is now a Ph.D. candidate at Division of Product Development and Technology, Faculty of Agro-Industry, Chiang Mai University. The research specialties were Management in Agro-industry, Product development technology, Experimental design, Sensory evaluation principle, Gas Chromatography technology, Aroma and fragrance release, Encapsulation and Electronic nose. His publications are as follows:

R. Samakradhamrongthai, N. Utama-ang, and P. Thakeow, "Identification of volatile compounds released from dry scented Thai flowers and their potential application in flower-mixed tea," *Asian Journal of Food and Agro-Industry*, vol. 2, no. 4, pp. 525-534, 2009.

R. Samakradhamrongthai, P. Thakeow, P. Kopermsub, S. Chansakoaw, and N. Utama-ang, "Sensory acceptance and antioxidant activity of selected Thai aromatic plants," in *Proc. 4th International Conference on Natural Products for Health and Beauty*, Chiang Mai, Thailand, November 28-30, 2012.

Niramon Utama-ang was born in Phisanulok, Thailand on May 11, 1966. She received her B.S. degree in food science and technology from Chiang Mai University, Thailand in 1987, M.S. and Ph.D. degree in Agro-industrial Product Development form Kasetsart University, Thailand in 2006. Now, she is a lecture in Division of Product Development Technology, Faculty of Agro-industry, Chiang Mai University, Thailand. Her research area is in functional food product development, sensory and consumer test, herbal tea and rice products. Her publications are as follows:

N. Laokuldilok, P. Thakeow, P. Kopermsub, and N. Utama-ang, "Optimization of microencapsulation of turmeric extract for masking volatile flavors," *Food Chemistry*, vol. 194, pp. 695-704.

T. Boonchu and N. Utama-ang, "Optimization of extraction and microencapsulation of bioactive compounds from red grape (*Vitis vinifera* L.) pomace," *Journal of Food Science Technology*, vol. 52, no. 2, pp. 783-792, 2015.

S. Hirun, N. Utama-ang, Q. V. Vuong, and C. J. Scarlett, "Investigating the commercial microwave vacuum drying conditions on the physicochemical properties and radical scavenging ability of Thai green tea," *Drying Technology*, vol. 32, no. 1, pp. 47-54, 2014.

Associate Professor Dr. Utama-ang is the Deputy Director of Lanna Rice Research Center, Chiang Mai University, Thailand. Her research aims to develop Thai rice to be the kitchen of the world.