

# STUDIES ON DEVELOPMENT OF MAFAI JEEN [*Clausena lansium* (Lour.) Skeels] PRODUCTS

## INTRODUCTION

Mafai Jeen (*Clausena lansium* )Lour. (Skeels) is originally cultivated in the southern China and in the northern to central Viet Nam. It was introduced into Thailand by Chinese since 1897 (Anonymous, 2001), where it was predominantly grown in Nan province, the northern Thailand. Mafai Jeen fruit was known of having medicinal functions for stomach upset, indigestion and coughing (Stuart, 1977). In the Philippines, folkloric group used Mafai Jeen for influenza, colds and abdominal colic pains. All parts of Mafai Jeen were proved to be useful, the fruits were used as herb medicine for stomach upset and indigestion, the leaves were used as medicine for the treatment of coughing, asthma and gastro-intestinal diseases, the seeds were used for gastro-intestinal diseases such as acute and chronic gastro-intestinal inflammation, ulcers, etc. (Lin, 1989; Kan, 1972). The roots were used to treat bronchitis (Kan, 1972) and were used as an anti-malarial (Kong *et al.*, 1983).

In 2006, the cultivated area of Mafai Jeen in Nan province is 1,000 rais or 400 acres. Mafai Jeen fruits season is from begin of May to the end of July. The fresh Mafai Jeen must be preserved into other product, the pulp can be add to fruit cups, gelatins or other desserts, or made into pie, jam or jelly. Carbonated beverage resembling champagnes is made by fermenting the fruit with sugar (Morton, 1987), the most popular product is in dried form. Mafai Jeen dry preserve fruit, candy, wine, and juice products contain unique flavor and benefits to local medicinal treatment. The governor of Nan province proposed to promote dried Mafai Jeen product to be in the program of one tumbon one product (OTOP). At present in 2006, the fresh fruits are sold at a price of approximately 70 USD/ton.

## Objectives

The scope of this dissertation are:

1. To identify the aroma compounds of Mafai Jeen.
  - a. Aroma compounds of fruits, seeds, and leaves by headspace sampling.
  - b. Aroma compounds of Mafai Jeen fruits by HS-SPME analysis.
2. To study effect of drying methods on Mafai Jeen.
  - a. Effect of drying conditions on qualities of dried Mafai Jeen.
  - b. Aroma components of Mafai Jeen fruit treated by different drying condition.
3. To study the intensity of astringent taste of Mafai Jeen.
4. To develop Mafai Jeen candy with shelf life study.

The major contribution of this dissertation is the development of Mafai Jeen candy product and study on shelf life. Additional contributions include, the identification of aroma compounds of Mafai Jeen for further use and study the effect of drying methods on Mafai Jeen. Finally, study the intensity of astringent taste of Mafai Jeen. These contributions can be seen from the contents and will be summarized in the final conclusions.

## LITERATURE REVIEW

### 1. Mafai Jeen

Mafai Jeen (*Clausena lansium* (Lour.) Skeels) is a minor member of the Rutaceae and a distant relative of citrus fruits, it travelled sufficiently to acquire many vernacular names, mostly derived from the Chinese *huang-p'i-kuo*, *huang p'i ho*, *huang p'i kan*, or *huang-p'i-tzu*. In Malaya, it is known as *wampi*, *wampoi*, or *wang-pei*; in the Philippines, *uampi*, *uampit*, *huampit* or *galumpi*; in Vietnam, *hong bi*, or *hoang bi*. In Thailand, it is called *Mafai Jeen* (มะขามป้อม), and the common English name is wampee (see appendix Figure A1). In this dissertation, the name Mafai Jeen will be used. It is a highly valued backyard tree in southern China and Southeast Asia. It is an attractive tree with grapelike fruits, but the pulp is scant and the seeds is large (Morton, 1987).

Mafai jeen fruits has outside appearance alike regular Mafai fruits (*Baccaurea ramiflora* Lour.) harvesting in May to July for it season. Mafai Jeen is a native and commonly cultivated in southern China and the northern part to Central Vietnam. It is introduced to Thailand with Chinese immigration in 1897. Mafai Jeen is grown only in Nan province, especially Meung Nan district and nearby Nan river. Mafai Jeen is a second economic fruits of Nan province after Sri-Thong orange (*Citrus reticulata*). Mafai Jeen was growing in the Philippines before 1837 and was reintroduced in 1912. In India and Ceylon, Mafai Jeen was grow in a small area. Chinese people in southern Malaya, Singapore and elsewhere in the Malaysian Archipelago grow the tree at their backyard. It is cultivated to a limited extent in Queensland, Australia and Hawaii. In 1908, the record showed the growing in a few Hawaiian gardens for many years but was not in general cultivation. It was brought to Florida as an unidentified species in 1908. The United States Department of Agriculture received seed from Hong Kong in 1914; from Canton in 1917 and from Hawaii in 1922. Mafai Jeen tree was grown by Dr. David Fairchild at 'Kampong' in Coconut Grove, Miami, and a small cottage near it was named the 'Wamperi' (Morton, 1987).

The Mafai Jeen trees are medium shrub, evergreen habit and can grow up to 10 meters in height. The tree can be grown either fast or slow, depending on its physiology and environments, flexible branches, and gray-brown bark rough to the touch. The leaves are dark green ruffled pinnate compound consisting of 4-7 leaflets, elliptic or elliptic-ovate leaflets  $2\frac{3}{4}$  to 4 inch (7-10 cm) long, oblique at the base, waxy-margined and shallowly toothed; thin, minutely hairy on the veins above and with yellow, warty midrib prominent on the underside. The petiole also is warty and hairy. The inflorescence is a many branched panicle. It bears hundreds of small whitish or yellowish flowers which give off a mild, pleasant odor when the tree is in full bloom. The number of fruit maturing on a panicle may vary from 1 to 30, or more, hairy panicles 4 to 20 inch (10-50 cm) long. The fruits, on  $\frac{1}{4}$  to  $\frac{1}{2}$  inch (0.6-1.25 cm) stalks, hang in showy, loose clusters of several strands. The Mafai Jeen fruits may be round, or conical-oblong, up to 1 inch (2.5 cm) long, with 5 faint, pale ridges extending a short distance down from the apex. The thin, pliable but tough rind is light brownish-yellow. It is easily peeled and too resinous to be eaten. The flesh, faintly divided into 5 segments, minutely hairy and dotted with tiny, brown oil glands. They are very juicy with a translucent pulp similar to a grape. The fruits are highly aromatic, the taste has a good deal of the grapelike fruits, accompanied with a peculiar flavor, being very grateful to the palate, mucilaginous, juicy, pleasantly sweet, subacid, or sour depending on the variety and ripeness. There may be 1 to 5 oblong, thick seeds  $\frac{1}{2}$  to  $\frac{5}{8}$  inch (1.25-1.6 cm) long, bright-green with one brown tip. The fruit is borne in clusters, resembling, when ripe a diminutive lemon. It contains 1 to 3 seeds which nearly fill the interior. We eat with a peel as same as kamquat. Florida-grown fruits have shown 28.8 to 29.2 mg/100 g ascorbic acid (Morton,1987).

Mafai Jeen grown in Thailand was divided into 2 different varieties. 1) Sweet-sour flavor; round shape or chicken-heart and brown skin. 2) Sweet flavor; conical-oblong shape or long-chicken-heart and yellow-skinned.

Mafai Jeen cultivation is done in the similar procedure as for the other citrus trees. A sunny, well drained site with plenty of water and organic matter should be

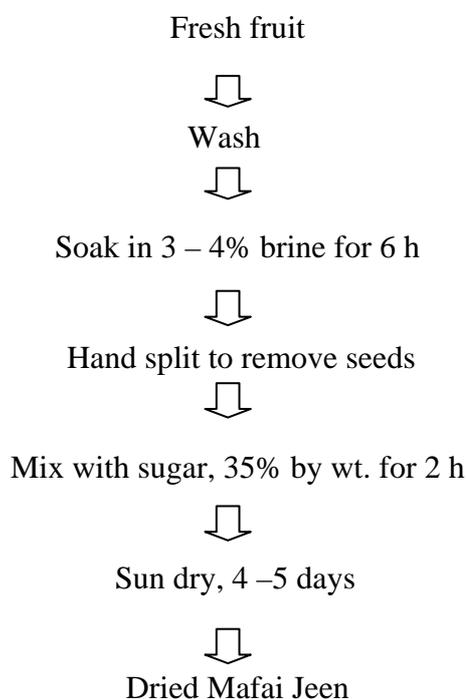
managed for these trees. The crop is borne solely on the tips of branches, so the less pruning the better production. Very few problems have been observed with pest and diseases other than occasional infestations of aphids. The fruits ripen in May to July in Nan province. Mature trees may yield 100 kg of fruits in a season. A grafted Mafai Jeen will produce fruit in around 3-4 years. A fully ripe, peeled Mafai Jeen, of the sweet or subacid types, is agreeable to eat out-of-hand, discarding the large seed. The fruit pulp can be added to fruit cups, gelatins or other desserts, or made into pie or jam. Jelly can be made only from the acid types when under-ripe. The Chinese serve the seeded fruits with meat dishes. In Southeast Asia, carbonated beverage resembling champagne is made by fermenting the fruit with sugar and straining off the juice, but the most popular product is dried Mafai Jeen. The process method are secret they known only the Chinese group.

Dried Mafai Jeen were originated in China. Mafai Jeen fruits were dried with pericarp and heating was introduced in this process become the product with dark in color. The dried Mafai Jeen fruits can be kept for a long time. The process of drying Mafai Jeen fruits can be done by following method show in [Figure 1](#).

## **2. Medicinal Uses**

The fruit was used as a medicine for stomach upsets, indigestion, and coughs. Folkloric uses in the Philippines were for influenza, colds and abdominal colic pains (Stuart, 1977). The leaves have been used as a folk medicine for the treatment of coughs, asthma and gastro-intestinal diseases, while the fruits are used for digestive disorders, and the seeds for gastro-intestinal diseases such as acute and chronic gastro-intestinal inflammation, ulcers, etc (Lin, 1989; Kan, 1972). Its roots are used in folk medicine to treat bronchitis (Kan, 1972) and as an anti-malarial (Kong *et al.*, 1983). The leaves, roots and fruits have been used as a folk medicine in Taiwan (Li *et al.*, 1991) and China (Yang *et al.*, 1988) for treatment of certain dermatological diseases such as, for instance, acute and chronic viral hepatitis.

### Dried Mafai Jeen process



**Figure 1** Dried Mafai Jeen process

Source: Anonymous (2001)

Eating Mafai Jeen will counteract the bad effects of eaten too many lychees. Lychees should be eaten when one is hungry, and Mafai Jeen only on a full stomach.

The halved, sun-dried, immature fruit is a Vietnamese and Chinese remedy for bronchitis. Thin slices of the dried roots are sold in Oriental pharmacies for the same purpose. Indidactone compound was found in the Mafai Jeen leaf. The leaf decoction is used as a hair wash to remove dandruff and preserve the color of the hair. (Perry, 1980)

Three novel cyclic amides was isolated from the leaves of *C. lansium*, the boiled water extract of its leaves is used in folk medicine for the treatment of certain dermatological diseases and of acute and chronic viral hepatitis. Heptaphylline and

dehydroindicolactone and lansamide-I have been isolated from the roots and leaves of the plant (Yang *et al.*, 1988).

Cinnamamide derivatives was extracted from the seed (Lin, 1989).

The characterization of dehydroindicolactone (Kong *et al.*, 1983) and coumarins (Kumar *et al.*, 1995) from the root bark; Carbazole alkaloids was isolated from the root (Li *et al.*, 1991).

The homodimeric trypsin inhibitor with a molecular mass of 54 kDa was isolated from the seed (Ng *et al.*, 2003). It was found to possess antifungal and HIV reverse transcriptase-inhibitory activity.

### **3. Drying**

Food drying is one of the oldest methods of preserving food for later use. It is a complex operation involving heat and mass transfer which may cause changes in product qualities. Physical changes that may occur included shrinkage, puffing and crystallization. In some cases, desirable or undesirable chemical or biochemical reactions may occur leading to changes in color, texture, odor or other properties of the food product (Maskan *et al.*, 2002). Drying occurs by vaporization of the liquid by supplying heat to the wet material. Heat may be supplied by conduction (contact or indirect dryers), by convection (direct dryers), by radiation or volumetrically by placing the wet material in a microwave or radio frequency electromagnetic field. Over 85% of industrial dryers are of convection type with hot air or direct combustion gases as the drying medium (Devahastin, 2000). Drying of agricultural products has always been of great importance to the preservation of food by human being. Sun drying is still the most common method used to preserve agricultural products in most tropical and subtropical countries (Yaldiz *et al.*, 2001).

#### **4. Aroma Compounds**

Aroma or volatile compounds are the most sensitive components in the process of food drying. The effect of drying on the composition of volatile constituents of various aromatic plants and vegetables has been the subject of numerous studies, which show that the changes in concentrations of the volatile compounds during drying depend on several factors, such as the drying method and parameters that are characteristic of the product subjected to drying (Venskutonis, 1997).

Volatile compounds must be isolated and concentrated to suitable concentration prior to analysis by Gas Chromatography. Essential oils and aqueous sample can be easily directly injected after diluting or concentrating. For complex food samples, extraction technique is the most appropriate. Extraction techniques for aroma in food are based on two different properties of their volatile constituents. They are volatility and affinity toward non-polar solvents or polymer. Distillation and headspace trapping use volatility to selectively remove volatile components from food matrix. Solvent extractions and adsorptions polymers use their hydrophobicity for the same purpose (Etievant, 1996). The extraction method must be represented the original flavor present in the matrix and avoided the formation of any artifact during sample preparation (Chaintreau, 1999). Chaintreau (2001) classified the extraction methods into three main categories (Table 1). Various extraction methods have been widely used for the analysis of volatile components of fruits.

##### 4.1 Distillation

Several techniques of distillations have been used to extract aroma components from foods. Steam distillation is among the oldest techniques used to separate volatile from non-volatile material. Aroma extracts can be obtained very fast and simply by steam distillation.

##### 4.2 Headspace Extraction

Steam distillation, solvent extraction or solid phase extraction (SPE) techniques allow quantitative data to be obtained, but are often labor-intensive

Table 1 Main extraction methods of aroma compounds.

Solubility	Volatility	Miscellaneous
-Soxhlet	-Steam distillation	-Simultaneous distillation-
-Liquid-liquid extraction	-Vacuum transfer	extraction (SDE)
-Supercritical fluid extraction (SFE)	-Headspace (HS)	-Distillation-membrane extraction
-Solid phase extraction (SPE)		-Simultaneous distillation- adsorption
-Solid phase micro- extraction (SPME)		

Source: Chaintreau (2001)

(Dumont and Adda, 1979; Nijssen, 1991; Saxby, 1982). Besides, chromatographic signals of trace substances may be obscured by high concentrations of low-volatile compounds (Bonino *et al.*, 2003). Headspace analysis may overcome these disadvantages, allowing analysis of the volatile fraction only.

Headspace extraction is the method for monitoring the gaseous headspace above a liquid or solid in a sealed container and suitable for aroma analysis. It is a non-destructive technique of mild conditions and easy sample preparation. This technique is divided into three broad categories: static headspace, dynamic headspace, and purge & trap. The fundamental principle of each technique is the same. Volatile analyzes from a solid or liquid material are sampled from the atmosphere adjacent to the sample. In static headspace techniques, a small sample of the atmosphere is sampled and injected directly onto a GC column. In dynamic headspace techniques, the organic analyzes from the headspace are first concentrated and then transferred to GC. The term dynamic headspace is usually used when refer to the analysis of solid materials and the term purge & trap generally refers to the

analysis of liquid samples by bubbling the purge gas through system (Wampler, 1997). The gas flow entrains the analytes on to adsorbent trap, where they remain until the trap is heated to desorb the analytes into the carrier gas stream (Hinshaw, 2000). Dynamic headspace extraction involves the adsorption of organic molecules swept by an inert gas on an organic polymer. There are many types of available polymers such as Tenax, Porapak, Chromosorb, and charcoal. The trapping step may involve adsorption onto a high surface sorbent material or cold trapping by condensing or freezing (Wampler, 1997)

Among headspace sampling techniques, headspace solid phase microextraction (HS-SPME) has specific advantages over conventional static, dynamic and purge and trap techniques: it is economic, faster and requires little manipulation of the sample (Elmore *et al.*, 1997; Jelen *et al.*, 1998; Stevenson and Chen, 1996; Xiaogen and Pepard, 1994).

Solid-phase micro-extraction (SPME) is an innovative, completely solvent free sample preparation method. It can be used to concentrate flavors and fragrances that significantly reduced the time and cost required for sample preparation. This technique can be used to concentrate volatile organic compounds from liquid samples by immersion SPME or headspace above a liquid or solid sample by HP-SPME. The analytes from sample are concentrated on fused silica fiber coated with a polymer film. The equilibrium distribution of analytes is established between the stationary phase (the microfibers) and the aqueous or gas phase (sample). Once equilibrium has been established, the concentrated compounds are thermally desorbed in the injector of a gas chromatograph and transfer to the capillary column. The different polar phase films for SPME fibers are available for extracting organic compounds from various sample matrices e.g. PDMS (polydimethylsilosane), PA (polyacrylate), PDMS/DVB (polydimethylsilosane/divinylbenzene), CW/DVB (carbowax/ divinylbenzene), Carboxen-PDMS (carboxen-polydimethylsilosane), and DVB-carboxen (divinyl-benzene/carboxen) (Werkhoff *et al.*, 2002). The accuracy of SPME for quantitative analysis is highly dependent on experimental conditions,

sample matrix, analyze characteristics, the type of fibers and calibration techniques (Hinshaw, 2000 and Marsili, 2002).

SPME has been used for the analysis of a wide range of food products, such as; coffee (Bicchi *et al.*, 1997; Ryan *et al.*, 2004 ), cheese (Chin *et al.*, 1996), beef (Soo–Yeun and Eunice, 2004), pork (Elmore *et al.*, 2001; Fernando *et al.*, 2003), sausages (Marco *et al.*, 2004), hops (Field *et al.*, 1996), cinnamon (Miller *et al.*, 1996), wine and other alcoholic beverages (Evans *et al.*, 1997; Fischer and Fischer, 1997; Gandini and Riguzzi, 1997; Ong and Acree, 1999; Sala *et al.*, 2002; Demyttenaere *et al.*, 2003 ), apple (Matich *et al.*, 1996; Jun-Song *et al.*, 1997; Barbara *et al.*, 2004), and strawberry (Ulrich *et al.*, 1997).

Since its introduction by Arthur and Pawliszyn (1990), the technique of solid phase microextraction (SPME) has been widely used for the extraction and pre-concentration of an extensive range of analytes in a variety of samples. Flavors and fragrances are samples for which SPME has been extensively used.

The application of SPME coupled to GC–MS to detect and quantify aroma-related heterocyclic compounds generated by the Maillard reaction was addressed by Coleman III (1997) with detection limits as low as  $\mu\text{m l}^{-1}$ . HS–SPME was also applied to determine sulfur-containing compounds in onion (Jarvenpaa *et al.*, 1998) and wine aromas (Mestres *et al.*, 1998). Several fruit juices and pulp aromas also have been studied with SPME coupled to GC: orange (Steffen and Pawliszyn, 1996), tomato and strawberry (Song *et al.*, 1998), avocado (Lopez *et al.*, 2004) and mango (Malundo *et al.*, 1997), among others.

Previous studies on *C. lansium* included the characterization of many compounds from the roots, seeds, and leaves. However, despite the many studies that have elucidated the non-volatile composition, there is little research on the volatile compounds responsible for the intense aroma of Mafai Jeen, although Zhao *et al.* (2004) analyzed the essential oil of the leaf, flower, sarcocarp and seed of

*C. lansium*. The dominant constituents identified were  $\beta$ -santalol,  $\alpha$ -santalol, methyl santalol, bisabolol and ledol.

## 5. Gas Chromatography-Mass Spectrometry (GC-MS) Analysis

Gas chromatography is the separation technique of a mixture of volatile components, following vaporization in an injection port and transfer into a narrow bore capillary column (typically, 0.25-0.32 mm i.d.). Separation is occurred by differential portion of analyzes between an inert carrier gas, such as helium, which is continuously flowing through the column and polymeric stationary phase (of selected polarity). The inside of the column is coated or chemically bound. The column is housed in a temperature programmed. Then, the analyzes are detected sequentially as they elute from the column by detector (Peppard, 1999). Only those materials that can be vaporized without decomposition are suitable for GC (Kitson *et al.*, 1996)

GC-MS is the combination of two powerful analytical techniques. The gas chromatograph separates the components of mixture in time and the mass spectrometer provides information that aids in the structural identification of each component (Kitson *et al.*, 1996). Flavor extracts are separated into their individual components by GC and a characteristic mass spectrometer. Individual components of mixtures are identified by comparing their mass spectra (and GC retention time or indices) to those of authentic compounds. Different molecules can be broken up into fragments in a consistent and repeatable manner when subjected to high-energy electron bombardment (typically 70 eV). It occurs within the ion source of a mass spectrometer operated in electron impact mode. The results of electrically charged fragment may then be separated according to their mass/charge ( $m/z$ ) ratio; subsequent detection of this ions yields a mass spectrum characteristic of the original molecule. It is depended on the type of compound involved. In addition, mass spectra may be searched against huge libraries of spectra, including commercial and much more important in the case of many flavor analysis laboratory and proprietary libraries (Peppard, 1999).

## 6. Astringency

Astringency, chemically defined as the ability to precipitate proteins (Bate-Smith, 1954), is often described as dryness, puckering and rough-mouthfeel. The sensation of astringency is often associated with polyphenolic substances (Joslyn and Goldstein, 1964), which also elicit bitterness (Robichaud and Noble, 1990). Although sourness is the major sensation of organic acids, dryness or astringency of acids has also been reported (Rubico and McDaniel, 1992). Generally astringency is considered to be a tactile rather than gustatory stimulus, as recently illustrated by Breslin *et al.* (1993). However, interaction with gustatory stimuli has been shown to affect perception of astringency. Raising acidity (and perceived sourness) increased astringent intensity (Guinard *et al.*, 1986) and duration (Fischer, 1990). Both mouth dryness and bitterness of tannic acid were attenuated by the addition of sweeteners to test stimuli (Lyman and Green, 1990). Similarly, the astringency elicited by alum was decreased by sucrose (Breslin *et al.*, 1993)

The astringency belongs to mouthfeel sensations, particularly important in beverages, such as fruit juices, tea or wine. The sensory evaluation of astringency does not give a satisfactory information on the sensation as the sensation of astringent taste lasts relatively long time even after swallowing the draught. Therefore, time-intensity technique was applied for the measurement of astringency ( Lee and Lawless, 1991 ). Time-intensity (TI) is a descriptive sensory technique in which the intensity of one or more sensory characteristics is rated in real time. TI has been widely used as a tool to study the dynamics of flavor release (Piggott *et al.*, 1998) The TI procedure was found better than the use of category scales ( Lundahl, 1992 ) The application of TI procedure was found particularly useful in evaluation of astringency and bitterness of wine ( Noble, 1995 ). Trained judges rated the astringency and bitterness of catechin, gallic acid, grape seed tannins and tannic acid in white wine ( Robichand and Noble, 1990 ). The selection and training of panelists for TI requires understanding of complex abstract concepts and well adapted motor skills. Accepted training methods identify suitable panelists as those who can focus on

a single sensory attribute, and record changes in sensation as they occur (ASTM, 1998).

## **7. Sensory Analysis**

Sensory analysis is a scientific discipline used to evoke, measure, analyze, and interpret reactions to characteristics of foods and materials perceived by the senses of sight, smell, taste, touch and hearing (Meilgaard *et al.*, 1999). Sensory analysis methods are used in quality control, product development, storage change on the perceived sensory properties of food products, marketing research, and development applications. Sensory analysis provides marketers with an understanding of food product quality, directions for product quality, profiles of competing products, and evaluations of product reformulations from a consumer perspective (Stone and Sidel, 1993). The primary goal of sensory analysis is to conduct valid and reliable tests in producing data for which important and sound decisions can be made (Meilgaard *et al.*, 1999). Lawless and Heymann (1999) identified the two primary areas of sensory analysis to be analytical and affective tests.

Analytical tests consist of discrimination tests, threshold determination, and descriptive analysis (Lawless and Heymann, 1999; Meilgaard *et al.*, 1999). Discrimination tests consist of three different sub-categories all of which are based on the perceived differences between two or more food products, e.g., paired-comparison, triangle testing, and duo-trio testing (Stone and Sidel, 1993; Lawless and Heymann, 1999). Discrimination tests are to be used when there is a slight or minimal difference between samples (Chambers and Wolf, 1996) and is applicable in product reformulation, product positioning, ingredient changes, and cost reduction changes (Chambers and Wolf, 1996; Marketing Research Methodological Foundations, 2003). Threshold testing is a method to determine the strength or concentration of a stimulus required to produce effects on four different levels (Chambers and Wolf, 1996). The four different levels include detection threshold, recognition threshold, difference threshold, and terminal threshold (Chambers and Wolf, 1996). These methods are used in determining product acceptability, detecting product

contaminants, and to assist in product formulation (Chambers and Wolf, 1996; Stone and Sidel, 1993). Descriptive analysis has been widely used to characterize in detail aroma, flavor, and oral texture attributes of food products. Descriptive analysis is the description of both qualitative and quantitative sensory aspects of a product using trained panelists (Meilgaard *et al.*, 1999). Qualitative aspects involve selecting the characteristics in a product (appearance, flavor, aroma and/or texture). Quantitative aspects involve intensity ratings of the characteristics of a product. The panelists are used as an instrumentation source. Panelists are screened, selected, (approximately 6-15 people), and then trained. Descriptive panels usually require 50-100 hours of training prior to collecting and using panel data (Meilgaard *et al.*, 1999). After an extensive training, panelists have the expertise to evaluate aspects of a food product qualitatively and quantitatively. Descriptive sensory techniques include Quantitative Descriptive Analysis (QDA<sup>®</sup>), Flavor Profile Analysis (FPA), Texture Profile Analysis (TPA), Spectrum<sup>™</sup> Descriptive Analysis, Free Choice Profiling, and Time-Intensity Descriptive Analysis.

Affective tests can help the sensory scientist to understand the behavior of different consumers groups (Piggott, 1988), and therefore to understand potential buyers of the product and in which way such a product can be inserted into the food market. Data obtained from consumer affective tests represent key information in studies of product development, quality control, food product acceptance, and food service evaluation (Piggott, 1988). There are two types of affective tests, quantitative and qualitative. Qualitative tests (i.e., focus group interviews, focus panel, one-on-one interviews) measure subjective responses of a small group of representative consumers to the sensory properties of products by having them talk about their feelings in an interview or group setting (Meilgaard *et al.*, 1999). Quantitative tests consist of preference tests and acceptance tests, determine the responses of a large group of consumers to a set of questions regarding preference, liking, sensory attributes, etc. (Meilgaard *et al.*, 1999). Affective tests typically use consumers or untrained panelists for a particular product evaluation.

Food companies regularly use sensory tests, such as descriptive analysis and consumer affective tests, to study ingredient effects, processing variables and storage changes on the perceived sensory properties of their products.

## **8. Shelf Life**

In the product development of any food product, one considerable aspect is the knowledge of the shelf life. Shelf life is defined as the period between manufacture and retail purchase of a food product during which the product is of satisfactory quality (IFT, 1974). It can be defined as the time between the production and packaging of the product and the point at which it becomes unacceptable under given conditions (Ellis, 1994).

The Institute of Food Technologists' Expert Panel on Food Safety and Nutrition (IFT, 1981) indicated dried foods to be shelf-stable foods. Development of foods with long shelf life normally requires data on shelf life of the product in a shorter time than it takes for the full shelf life study in order to meet product launch schedules. Accelerated shelf life testing (ASLT) procedures are often used. To achieve results in a shorter time-frame, accelerated shelf life testing implies any method that is capable of evaluating product stability, based on data that is obtained in a significantly shorter period than the shelf life of the product (Mizrahi, 2000). Storage studies to predict shelf life are an important part of every product development (Dethmers, 1979; Waletzko and Labuza, 1976). It is also often assumed that accelerated deterioration can be achieved by raising the storage temperature, using an Arrhenius model (Labuza and Schmidl, 1985). The shelf life plots are useful in incorporating the effect of temperature on changes in food quality (Singh, 2000).

Chemical, physical and microbiological changes may occur during storage and result in a decrease of the sensory qualities of a food product (Labuza and Schmidl, 1985; Singh, 2000; Blackburn, 2000). At favorable temperature, such as room temperature, many enzymatic reactions proceed at rapid rates altering the quality attributes of foods (Singh, 1994). Color is one important of food quality that can be

used for analyses of quality changes as a result of storage (Giese, 2000). Color deterioration has been reported as follow a first-order reaction (Steet and Tong, 1996). Changes in all the different sensory characteristics: appearance, odor, flavor and texture changes can occur all though the shelf life of foods.

According to Labuza and Riboh (1982), shelf life is not a function of time alone, rather it is a function of the environmental conditions and the amount of quality change can be allowed. The environmental conditions often relate to the temperature of food products during storage and distribution (Labuza and Szybist, 1999). Physical, chemical and sensory quality changes of shelf-stable foods are roughly proportional to their storage temperature (IFT, 1974). The effect of temperature on the rates of chemical reactions were studies in many recent researches (Trezza and Krochta, 2000; Ahmed and Shivhare, 2001; Ahmed and Shivhare, 2002; Cardelli and Labuza, 2001; Lee and Krochta, 2002; Gills and Resurreccion, 2000; Grosso and Resurreccion, 2002; Uddin *et al.*, 2002). To determine shelf life, either consumer acceptance or trained panel assessment can be used (Dethmers, 1979; Ellis, 1994; Kilcast, 2000; Cardelli and Labuza, 2001). Resurreccion (1998) stated that acceptance tests are necessary to obtain the shelf life of a product. Several shelf life studies used consumer acceptance to determine the end of shelf life (Dethmers, 1979; Duyvesteyn *et al.*, 2001; Cardelli and Labuza, 2001; Grosso and Resurreccion, 2002; Hough *et al.*, 2002).

## **9. Fruit Candy**

Fruit candy is made primarily of sugar, glucose syrup, and fruit puree. Sometimes an additional natural thickening agent is used in order to improve smoothness and prevent separation. The qualities of fruit candy depends mainly upon ingredients used in the mix, which include water, sugar, glucose syrup, fruit, and a thickening agent. Ingredients interact with each other and create a delightful taste. The thickening agents play an important role in the production of fruit candy, improve product consistency and stabilization, reduce the number of crystals or the size of crystals or both during the production period and also retard melting at the

consumption stage. Their primary purpose is to produce smoothness in body and texture, reduce crystal growth during storage, extend shelf life, and provide uniformity of product and resistance to melting (University of Guelph, 2005). Polysaccharide gums are used widely as thickening agents in food.

## MATERIALS AND METHODS

### 1. To Identify the Aroma Compounds of Mafai Jeen

#### 1.1 The Aroma Components of Fruits, Seeds, and Leaves by Headspace Sampling

The objective in this study was to determine the headspace volatile components of Mafai Jeen fruits, seeds and leaves to reveal the volatile compounds that are responsible for its intense aromatic profile.

##### 1.1.1 Plant Materials

The fruits, seeds, and leaves of Mafai Jeen were collected in July 2004 from the Horticultural Research Station, Department of Agriculture, Nan Province, which is located in the northern part of Thailand. The plant (Forest Herbarium No. BKF 135985) was identified and deposited at the Forest Herbarium (BKF), National Park, Wildlife and Plant Conservation Department, Ministry of National Resources and Environment, Bangkok 10900 Thailand. All Mafai Jeen fruits used came from the same batch. Fully ripe fruit were used in the study; ripeness is determined when the fruit turns yellow and has a thin, sometimes brittle skin, somewhat like paper.

##### 1.1.2 Sample Preparation and Headspace Sampling

An Agilent 7694 (Agilent Technologies, Inc., Wilmington, DE 19808, USA.) was used for headspace sampling. Samples of 50 g were cut and immediately crushed in a blender, then 1 g of all samples used in this study were placed into 25 ml vials; they were then crimped and equilibrated for 20 min at 80 °C before headspace sampling, following the method of Alasalvar *et al.* (1999)

### 1.1.3 Gas Chromatography-Mass Spectrometry (GC-MS)

GC-MS was performed on an Agilent 6890 GC Plus equipped with a HP-5973 mass-selective detector (Agilent Technologies). A fused silica capillary column, HP-5-MS, with 5%-Phenyl methylpolysiloxane as non-polar stationary phase (30 m x 0.25 mm i.d. x 0.25  $\mu\text{m}$  film thickness, Agilent Technologies), was utilized for analysis of volatiles obtained from Mafai Jeen. The injection port temperature was 250 °C. The column temperature program started at 40 °C upon injection. The temperature was increased at a rate of 3 °C/min to 100 °C, and then at a rate of 5 °C/min to 230 °C, and held there for 2 min. Purified helium gas at a flow rate of 1 mL/min was used as the GC carrier gas. The mass spectrometer was operated in the electron impact (EI) mode with an electron energy of 70 eV; ion source temperature, 230 °C; quadrupole temperature, 150 °C; mass range  $m/z$  35-400; scan rate, 0.25 s/scan; EM voltage, 1423 V; and the GC-MS transfer line was set to 280 °C.

### 1.1.4 Qualitative and quantitative analyses

Most constituents Identification of volatile components was performed by matching their mass spectra with reference spectra in the Wiley 275 Mass Spectral Library (Revision C.00.00) and the National Institute of Standard and Technology (NIST) 98 Mass Spectral Library (Revision D.01.00/Search Program v.1.6d), both purchased from Agilent Technologies. Quantitative analysis of each volatile component in percent was performed by peak area normalization measurement.

## 1.2 Aroma Compounds of Mafai Jeen Fruits by HS-SPME Analysis

In this work, manual HS-SPME-GC-MS was employed to isolate and identify the main constituents of the aroma of Mafai Jeen fruits. The objective of this study was to compare the sample preparation, i.e., PDMS, PA, and

DVB/CAR/PDMS, used for determination of the aroma compounds in Mafai Jeen fruit.

### 1.2.1 Fruit Pulp Sample

Fresh Mafai Jeen fruits were collected in May 2004 from Nan Province in northern Thailand. The sample was homogenized in a blender for ca. 2 min and the resulting slurry was immediately transferred to a 50-ml glass syringe, which had its tip sealed with a silicone cap, and was completely filled. The syringe containing the slurry was kept refrigerated at 8 °C when not in use to minimize the loss of the most volatile compounds (Augusto *et al.*, 2000).

### 1.2.2 SPME Fibers

Three SPME fiber coatings (Supelco, Bellefonte, PA, USA) were evaluated and used for the extraction procedures: 100 µm polydimethylsiloxane (PDMS, non-bonded) cat. No.57300-U, 85 µm polyacrylate (PA, bonded) cat. No. 57304, and 50/30 µm divinylbenzene/carboxen/PDMS (DVB/CAR/PDMS, bonded) cat. No.57328-U. Fibers were conditioned prior to use according to supplier's prescriptions and the contaminated fiber cleaned at 20 °C below its recommended maximum temperature for 1 h.

### 1.2.3 Headspace Extraction Procedure

In each extraction, 2.00±0.1 g of the pulp slurry was transferred from the syringe to a 40-ml clear screw top vial with hole cap PTFE/silicone septa (Supelco cat. No. 27089-U) and the system was kept at 50 °C with magnetic stirring to achieve the partition equilibration of the analysis between the sample and the headspace. After 15 min a SPME fiber was exposed to the headspace for 15 min. The sample transfer to the GC column was accomplished by keeping the SPME fibers for

5 min in the heat chromatograph injector. This headspace extraction procedure was performed in duplicate for each sample using the SPME fibers listed above. Blank runs with the used fibers were conducted between extractions, to check the absence of carry over which would cause memory effects and misinterpretation of results.

#### 1.2.4 GC–MS System

The extracts were analyzed with an Agilent 6890GC Plus equipped with a HP-5973 mass-selective detector (Agilent Technologies, Inc., Wilmington, DE 19808, USA.). A fused-silica capillary column, HP-5MS, with 5%-Phenyl methylpolysiloxane as nonpolar stationary phase (30 m x 0.25 mm i.d. x 0.25 $\mu$ m film thickness, Agilent Technologies) was utilized for analysis of volatile compounds and a split-splitless injector was operated in the splitless mode for all chromatographic runs.

The injection port temperature was 250 °C. The column temperature program started at 40 °C upon injection. The temperature was increased at a rate of 3 °C/min to 100 °C, and then at a rate of 5 °C/min to 220 °C, and held there for 2 min. High purity helium gas at a constant flow rate of 1 ml/min was used as the GC carrier gas. The mass spectrometer was operated in the electron impact (EI) mode with an electron energy of 70 eV; ion source temperature, 230 °C; quadrupole temperature, 150 °C; mass range  $m/z$  35-400; scan rate, 0.25 s/scan; Electron multiplier (EM) voltage was obtained from autotune, 1423 V. and the GC-MS transfer line was set to 280 °C. Compounds were identified by matching their mass spectra with reference spectra in the Wiley 275 Mass Spectral Library (Revision C.00.00) and the National Institute of Standard and Technology (NIST 98) Mass Spectral Search Program v.1.6d, both purchased from Agilent Technologies. Quantitative analysis of each volatile component in percent was performed by relative area normalization measurement.

## 2. To Study Effect of Drying Methods on Mafai Jeen

### 2.1 Effect of Drying Conditions on Qualities of Dried Mafai Jeen

The purpose of this study was to evaluate the effect of four drying methods; hot air drying at 60 °C, hot air drying at 45 °C, vacuum drying at 45 °C and sun drying, on qualities of dried Mafai Jeen fruits.

#### 2.1.1 Plant Material

The fresh Mafai Jeen fruits were collected in May 2004 from Nan Province located in the northern part of Thailand. The experiments were carried at Bangkok (13 ° 45 ' N, 100 ° 31 ' E) Thailand. All Mafai Jeen fruits used for experimental were from the same batch.

#### 2.1.2 Preparation of Dried Mafai Jeen

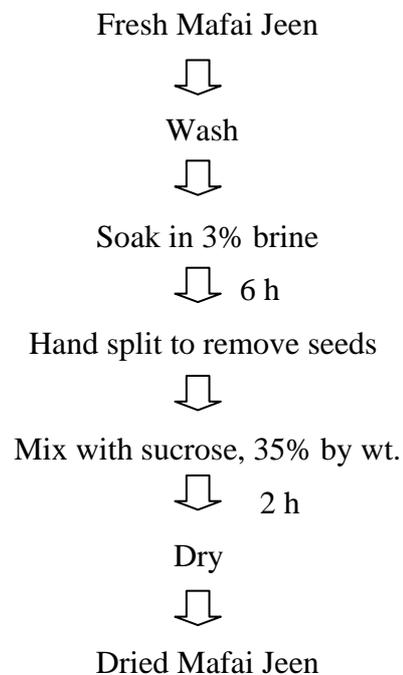


Figure 2 Flow chart of dried Mafai Jeen process.

Flow chart for dried Mafai Jeen production was shown in [Figure 2](#). The first step was washing of Mafai Jeen to remove dirt, leaves, and foreign materials. Then, the Mafai Jeen was soaked in 3% brine for 6 h. The fruits were separated from the branched panicle and deseeded manually. The flesh with peel was then mixed with 35% sucrose by weight and they were left for 2 h until sucrose impregnated as flesh followed by drying later.

### 2.1.3 Drying

The Mafai Jeen fruits were dried in a hot air dryer (BWS, B.W.S. Trading, Thailand. Overall dimensions: 1.92 m height, 1.22 m width, and 0.80 m depth), vacuum dryer (VD53, Binder, Germany. Overall dimensions: 0.75 m height, 0.63 m width, and 0.55 m depth), or sun-dried. The initial moisture content of the samples was 72% (wet basis). Drying conditions for hot air drying were temperature 45 °C or 60 °C, air flow 1.2-1.5 m/s, and tray load 4.00-4.50 kg/m<sup>2</sup>; for vacuum drying, temperature 45 °C and vacuum 300-400 mbar abs, and for sun drying, the samples were dried under direct sunlight, starting at 8:00 a.m. and continuing till 5:00 p.m., with temperature  $38.2 \pm 4.5$  °C, the per cent relative humidity of air  $52 \pm 6.2$ , and air velocity  $0.53 \pm 0.22$  m/s (these values are the average values,  $\pm$  standards deviation). Tray loading in vacuum and sun drying were kept the same as in hot air drying. Moisture loss was recorded for determination of drying curves by scale balance at 60 min intervals for hot air and sun drying and 120 min intervals for vacuum drying. The samples were dried until equilibrium was reached (no weight change).

### 2.1.4 Color Analysis

Color analysis was carried out on Mafai Jeen samples using a tristimulus colorimeter (Chromameter CR-200, Minolta, Osaka, Japan) to obtain the color values (CIE  $-L^*$   $a^*$   $b^*$  values).  $L^*$  represents lightness,  $a^*$  represents greenness (-) to redness (+) while  $b^*$  represents blueness (-) to yellowness (+) values

(Minolta, 1999). The changes in each individual color parameter were calculated as follows:

$$\Delta L^* = L^* - L_o^* \dots\dots\dots(1)$$

$$\Delta a^* = a^* - a_o^* \dots\dots\dots(2)$$

$$\Delta b^* = b^* - b_o^* \dots\dots\dots(3)$$

The subscript 'o' refers to the target value or the initial color parameters of each product at the beginning of the drying experiments. The total color difference ( $\Delta E$ ) was then determined using the following equation (Nsonzi and Ramaswamy, 1998):

$$\Delta E = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2} \dots\dots\dots(4)$$

At least fives measurements were carried out on each sample.

#### 2.1.5 Proximate Analysis

Proximate analysis were determined by the methods of Association of Official Analytical Chemists (A.O.A.C, 1990)

#### 2.1.6 Sensory Analysis

Preference tests of dried Mafai Jeen products were done with a 50 panel. All panelists were experienced with hedonic scale sensory test. The samples were randomized and coded with a three-digit number chosen from a table of random numbers. A 9-point hedonic scale (Meilgaard *et al.*, 1999) was used to assess the acceptance of various aspects (such as overall acceptance, appearance, aroma, flavor, and texture) of the products.

## 2.2 Aroma Components of Mafai Jeen Fruit Treated by Different Drying Condition

Dried whole Mafai Jeen is a unique product, consumed mainly in the northern part of Thailand. However, no reports were available on systematic drying of Mafai Jeen fruit, nor on the changes that occur in its aroma profile as a result of processing, though the fruit is valued mainly for its flavor and aroma. The objectives in this study are; to determine the changes in volatile aroma components of dried Mafai Jeen fruit, using different types of driers.

### 2.2.1 Plant Material

The fresh Mafai Jeen fruits were collected in May 2004 in Nan Province located in the northern part of Thailand. The initial samples were divided into five batches. One was stored frozen at  $-18^{\circ}\text{C}$  for fresh analysis. The remaining batches were dried by using one of the following different drying methods test: (a) sun drying at ambient temperature ( $30\text{-}39^{\circ}\text{C}$ ); (b) drying in a hot air oven at  $45^{\circ}\text{C}$ ; (c) drying in a hot air oven at  $60^{\circ}\text{C}$ ; (d) drying in a vacuum oven at  $45^{\circ}\text{C}$ . The samples were dried until moisture content reached approximately of 16% dry basis (calculation based on initial moisture content and weight loss,  $\text{mass of moisture/mass of dry solid} \times 100$ ).

### 2.2.2 Extraction of Volatiles

The Clevenger-type apparatus (Clevenger, 1928) hydro-distillation extraction was used for extraction and concentration of volatiles. The Mafai Jeen fruit samples, 500 g with fresh fruit or 200 g dried fruit was put into a round bottomed flask with 500 ml of distilled water. Five drops of silicone were added as antifoam. The distillation process was carried out for 6 h. The distillate was dried over anhydrous sodium sulfate. Dried distillate was concentrated under a stream of nitrogen and stored in 2 ml vial at  $-18^{\circ}\text{C}$  until analysis. The extraction of each sample to be submitted to gas chromatography-mass spectrometer (GC-MS) analyses.

### 2.2.3 GC-MS Analysis

A GC-MS (Agilent 6890 and HP 5973 mass-selective detector, Agilent Technologies, Inc., Wilmington, DE 19808, USA.) equipped with a fused silica capillary column, HP-5MS, with 5%-Phenyl methylpolysiloxane as non-polar stationary phase (30 m x 0.25 mm i.d. x 0.25  $\mu$ m film thickness, Agilent Technologies) was utilized for analysis of volatiles obtained from distillation of Mafai Jeen fruits. The samples (1  $\mu$ L) were injected with a split ratio of 10:1. The injection port temperature was 250 °C. The column temperature program started at 40 °C upon injection. The temperature was increased at a rate of 3 °C/min to 100 °C, and then increased at a rate of 5 °C/min to 230 °C, and hold for 2 min. Purified helium gas at a flow rate of 1 mL/min was used as the GC carrier gas. The mass spectrometer was operated in the electron impact (EI) mode with an electron energy of 70 eV; ion source temperature, 230 °C; quadrupole temperature, 150 °C; mass range  $m/z$  35-400; scan rate, 0.25 s/scan; EM voltage, 1423 V the GC-MS transfer line was set to 280 °C.

### 2.2.4 GC-MS Data Analysis

Identification of volatile components was performed by matching their mass spectra with reference spectra in the Wiley 275 Mass Spectral Library (Revision C.00.00) and the NIST 98 Mass Spectral Library (Revision D.01.00/1.6d), both purchased from Agilent Technologies. Quantitative analysis of each volatile component in percent was performed by peak area normalization measurement.

## **3. To Study the Intensity of Astringent Taste of Mafai Jeen**

The objectives in this study are; To explore the use of Time-intensity (TI) in the measurement of astringent taste, to study astringent taste in Mafai Jeen fruits and studied the effect of sucrose and ascorbic acid on the intensity of astringent taste.

### 3.1 Material

Tannic acid powder pure (Merck); citric acid, chemically pure for food use; sucrose, pure for food use; dried Mafai Jeen

### 3.2 Methods

The procedure used for the sensory analysis was in agreement with requirements of the international standard (ISO, 1985), performed in a standardized test room provided with 8 test booths. The room temperature varied between 20-25 °C, the relative moisture content between 40–70%

Seventeen volunteers (seven man and ten woman, ranging in age from 20–36 years) were chosen from the pool of staff at the Rajchamungala Institute of Technology, Nan campus, based on their lack of food allergies, tolerance of astringent flavors and availability on the training and sample testing days. Nine panelists were screened from this by their ability to rank order of differing taste content correctly. The training took place individually for each panelist over three sessions.

On the first session, the overall goals of the research project were described and panelist were given an overview of the procedure they would be carrying out. On the second and the third session of training, assessors were familiarized with astringent taste, and the technique of TI scaling especially aimed at evaluation of astringent taste. Then, they were asked to taste the standard reference of tannic acid solution 1 g/l. An unstructured line scale with descriptors ‘low astringency’ and ‘high astringency’, left to right respectively, 150 mm in length. The data were collected every 10 second.

Samples were presented in randomly 3 digit coded white plastic cups, not more than four samples were served in the random order, in interval of 2-3 min after

disappearance of astringent taste. Samples were evaluated in triplicate and presented at ambient temperature. Between sample, water and unsalted cracker were served for mouth washing.

Twelve samples of dried Mafai Jeen, varying in the rate of sucrose from 0, 10, 20 to 30 g/100g. of fruits and varying in the rate of ascorbic acid from 0, 1 to 2 g/100g. of fruits as shown in Table 2, were prepared by the following process;- The fresh fruits of Mafai Jeen in fully ripe, collected from the local orchard in Nan province were washed and soak in 4% of brine for 6 h, deseed by squeezing with hand, mix with sucrose and ascorbic acid, varying of concentration by treatment and holding for 2 h, drying with hot air dryer at 50 °C, for 36 h, and then store in cool dry place until the samples were use.

**Table 2** Composition of the experimental samples and reference standards.

	Sucrose (g / 100 g of fruits)	Ascorbic acid (g / 100 g of fruits)	Tannic acid (g / l)
Samples	0, 10, 20, 30	0	
	0, 10, 20, 30	1	
	0, 10, 20, 30	2	
Reference standard	0		1
solution	30		1

### 3.3 Data Analysis

Time to maximum intensity (T max), maximum intensity (I max), total duration (T tot) for astringency and sweetness were extracted from TI curves. Each variable was analyzed using SPSS 10.0 for Windows with a custom model analysis of variance in which the treatments and assessors were treated as a fixed factor. Mean astringency TI curve were calculated for low and for high flow subjects by averaging ratings across time and samples.

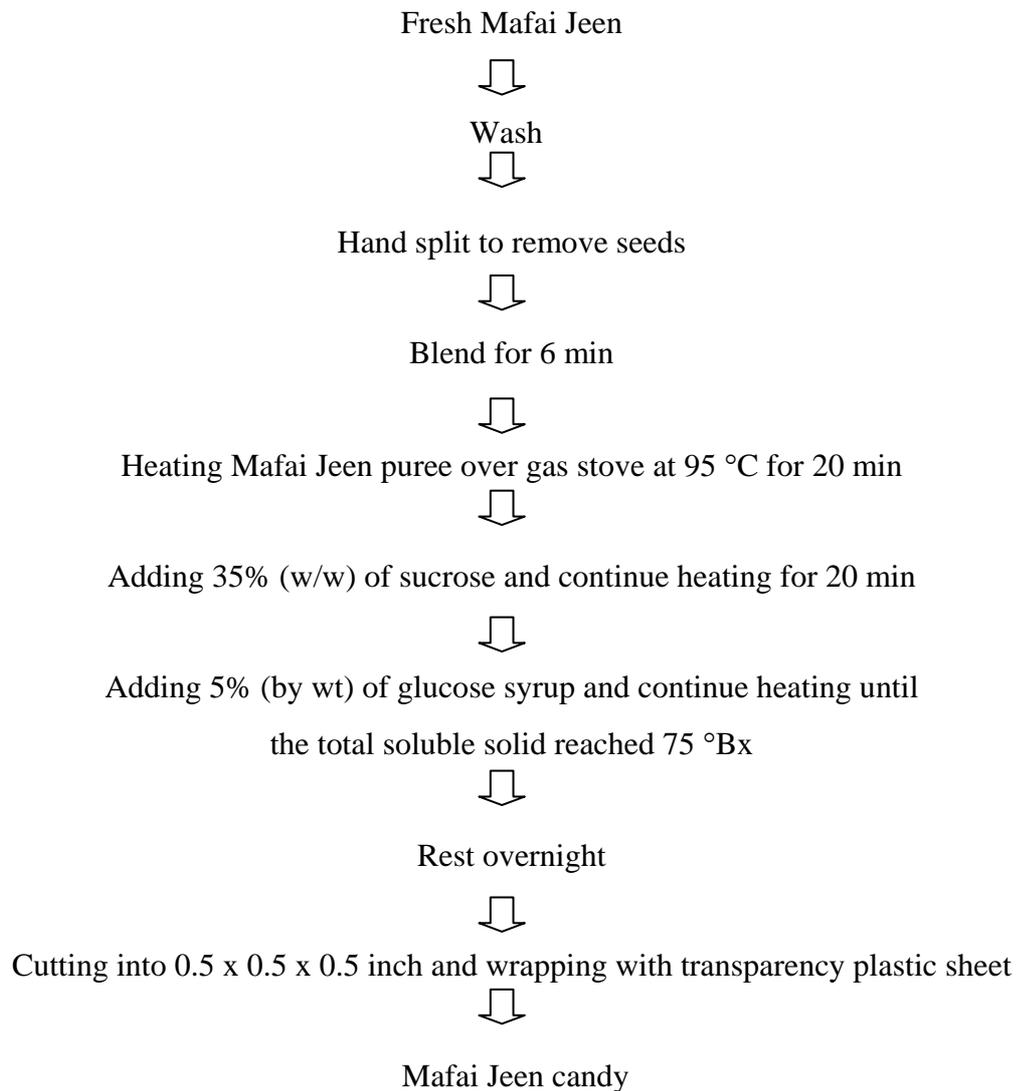
#### **4. To Develop Mafai Jeen Candy with Shelf Life Study**

##### 4.1 Determination of Mafai Jeen Qualities

The same batch of fresh Mafai Jeen fruits with similar ripening stage were collected from Nan province located in the northern part of Thailand. Physical and chemical determination and process development were done at the department of Product Development, Kasetsart University, Bangkok, Thailand. The Mafai Jeen fruits sample were remove seed before blending for 6 min and studied on qualities of color  $L^*$   $a^*$   $b^*$  values, pH, %moisture, %total soluble solid, %acidity, and %reducing sugar.

##### 4.2 Preparation of Mafai Jeen Candying

Flow sheet for Mafai Jeen candying production was shown in [Figure 3](#). The first step was washing of Mafai Jeen to remove dirt, leaves, and foreign materials. Then, the fruits were separated from branched panicle and deseeded manually. The flesh with peel obtained was blend for 6 minutes followed by heating Mafai Jeen puree over gas stove at 95 °C for 20 min, adding 35% (by wt) of sucrose and continue heating for 20 min, adding 5% (by wt) of glucose syrup and continue heating until the total soluble solid increasing to 75 °Bx. Samples obtained were rest overnight till it cool. Then, cutting into 0.5 x 0.5 x 0.5 inch and transparency plastic (oriented polypropylene, OPP film) was wrapping, thus obtained samples according to the candy process.



**Figure 3** Flow sheet of Mafai Jeen candy process.

### 4.3 Development of Mafai Jeen Candy

#### 4.3.1 Focus Group Discussion (FGDs)

Focus groups: a practical guide for applied research by Krueger and Casey (2000) was used as the basis for development of Mafai Jeen fruits candy instead of individual interviews because it was felt that participants would be more responsive and spontaneous in an informal. FGDs tend to facilitate considerable interaction on given topics in a limited space of time (Kidd & Parshall, 2000). Three

sessions of FGDs were conducted; two of the three sessions were 18-29 and 30-55 years old, held at Nan Province ( see appendix Figure C 3) whereas Mafai Jeen was grown, and one session was 18-29 years old, held in Bangkok ( see appendix Figure C 4). All FGDs were audio tape-recorded and lasted approximately between one to two hours. The sessions were held between 10 June 2005 and 20 August 2005. Groups were of mixed gender with 4–8 participants per group.

Data were collected in a systematic approach by asking semi-structured, open-ended questions and a socio-demographic questionnaire prior to commencing each focus group. This information complemented the discussion findings gained from the tape recorded sessions. Each focus group began with a general discussion on attitudes to foods, which allowed the respondents to relax and feel at ease. The questions were designed to explore issues related to implementation of the new product idea. The first series of questions were on Mafai Jeen fruit products selection, costs, packaging and benefits. And the second series of questions were on barriers to implementation and the suggestions for improving product and package.

#### 4.3.2 Study on Sucrose and Glucose Syrup Concentration in the Recipe

The aim of experiment was to examine the effect of sucrose concentration and glucose syrup concentration on physical and sensory acceptability of Mafai Jeen fruit candy. The experiment was conducted as a 3 x 5 factorial arrangement of treatment with 3 levels of glucose syrup concentration (0%, 5%, and 10% based on the total weight of the ingredients) and 5 levels of sucrose concentration (20%, 25%, 30%, 35%, and 40% based on the total weight of the ingredients, respectively). Mafai Jeen fruit candy were prepared follow as [Figure 3](#), sucrose were added after Mafai Jeen puree was heated for 20 min and continue stirring and heating for 20 min, then adding glucose syrup and continue stirring and heating until the total soluble solid increased to 75 °Bx and they were left over night, then cut the samples into 0.5 x 0.5 x 0.5 inches and wrapped with transparency plastic sheet. A total of fifteen Mafai Jeen fruit candy samples were made, each was prepared in duplicate (two experimental replication).

The texture profile characteristics of Mafai Jeen fruit candy were analyzed using the Lloyd Texture Analyzer (Lloyd TA 500, Intro Enterprise Co., Ltd. UK). The texture analyzer was equipped with a 50 kg load cell and a 50 mm diameter compression cell. The test speed was set at 50 mm/min and the distance to compress sample was 60% strain. The hardness (N) of the samples were recorded an standard of 10 measures was reported. (Vijayanand *et al.*, 2000)

The moisture content of Mafai Jeen fruit candy was determined by the methods of Association of Official Analytical Chemists (AOAC, 1990).

The water activity of Mafai Jeen fruit candy was determined at 25 °C using Thermoconstanter (Novasina, MLK, Switzerland), equipped with a temperature-controlled system which allow to have a temperature stable sampling environment. The equipment was calibrated with saturated salt solution in the  $a_w$  range of 0.43 – 0.75 (Favetto *et al.*, 1983). For each determination, five replication were obtained and the average reported; under these conditions reliability of this meter is about  $\pm 0.003 a_w$ .

Colorimetric measurement of Mafai Jeen fruit candy were determined in triplicate at the product surface using a spectrophotometer (CM 3500d, Minolta Camera Co., Ltd, Tokyo, Japan). The CIE –  $L^*$   $a^*$   $b^*$  uniform color space, where  $L^*$  indicates the lightness (0 = black, 100 = white),  $a^*$  indicates chromacity on a green (-) to red (+) axis, and  $b^*$  chromacity on a blue (-) to yellow (+) axis.

For consumer acceptance, two piece of each of the 15 Mafai Jeen fruit candy samples were evaluated by 30 untrained panelists. Panelists were asked to evaluate 3 sets of samples, each set composed of five samples and took a 10 min break between the set evaluated. They individually evaluated the samples in partitioned booths under fluorescent light at temperature 25 °C. A 9-points hedonic scale (1= dislike extremely; 5= neither like nor dislike; 9= like extremely) was used to

evaluate acceptability of the product attributes (color, taste, sweetness, texture, and overall acceptance).

The effect of white sugar concentration and glucose syrup concentration on physical and sensory acceptability of Mafai Jeen fruit candy were determined using a 3 x 5 factorial arrangement in completely randomized block design (RCBD) following the analysis of variance (ANOVA, SPSS version 10.0.1) method. Significant difference of treatment means were determined by Duncan's Multiple Range Test (DMRT). Statistical significant difference was established at  $p < 0.05$ .

#### 4.3.3 Study on Thickening Agent for Mafai Jeen Fruit Candy

Fruit candy is made of sugar, glucose syrup and fruit puree only, and sometime additional natural thickening agent will be used to improve the smoothness and prevent separation. The purpose of this study was to investigate by sensory evaluation an appropriate thickening agent for the Mafai Jeen fruit candy product.

Mafai Jeen fruit candy were prepared follow as [Figure 3](#), with concentration of sugar from the resulted of 4.3.2. A variety of thickening agents were tested in the study including; maltodextrin DE 10 (Staley, Tate & Lyle, USA), pectin (150grade /USA-SAG type B, Rapid set, GENU<sup>®</sup>, CP Kelco, Denmark ), and CMC (PM 7, China north chemical, China) with various concentration of 0.5%, 1.0%, 1.5%, 2.0%, and 2.5% (based on the total weight of the ingredients) were added simultaneous with glucose syrup during stir and then heating until the total soluble solid increasing to 75 °Bx and they were left for over night, then cut the samples into 0.5 x 0.5 x 0.5 inch and wrapped with transparency plastic sheet. The fifteen samples were evaluated by ranking of preference with 30 panelists. Each subject receive one type of thickening agent at a time, five samples coded with three-digit numbers and served in random order.

A non-parametric Friedman's test was performed to determine differences among the five samples. Fisher's analog of  $LSD_{rank}$  was applied to accomplish all paired-wise comparisons of the five Mafai Jeen fruit candy. The first rank in this study was used to study the sensory descriptive analysis.

The T value is calculated as follows:

$$T = \left\{ \left[ \frac{12}{bt(t+1)} \right] \cdot \sum X_{.j}^2 \right\} - 3b(t+1); j = 1 \text{ to } t \dots\dots\dots(5)$$

b = no. of panelists (blocks)

t = no. of samples

$X_{.j}^2$  = square of the rank sum of sample j

A two-tailed test

Ho: all sample are same

Ha: not all sample are same

At a specified  $\alpha$  level (0.05), if T value is greater than  $\chi^2(\alpha, df = t-1)$ , then reject

Ho. df = 4 when 5 samples tested.

If Ho is rejected, then do analog Fisher's LSD

$$\text{Analog Fisher's } LSD_{rank} = t(\alpha/2, \infty) \cdot \left[ \frac{bt(t+1)}{6} \right]^{1/2} \dots\dots\dots(6)$$

$t(\alpha/2, \infty)$  is the upper  $\alpha$  probability points of the student's t-distribution.

Two samples are declared to be significantly different at the  $\alpha$  level if their rank sum differs by more than the value of  $LSD_{rank}$

#### 4.3.4 Descriptive Sensory Analysis of Mafai Jeen Fruit Candy

##### a. Sample Description

First rank of preference of Mafai Jeen fruit candy added 3 type of thickening agent; maltodextrin, pectin, and CMC from 4.3.3 were used for descriptive sensory analysis. Each candy weighs  $3.5 \pm 0.23$  g the product was wrapped with transparency plastic sheet and packaged in an aluminum pouch for protection from light, humidity and foreign odors.

#### b. Panel Selection and Training

Fourteen panelists were recruited from Kasetsart University. They were pre-selected on the basis of good health conditions, time availability, no allergy to fruit candy products, interest in participating, available for all sessions, and able to verbally communicate about the product. Descriptive analysis test procedures as described by Meilgaard *et al.* (1999) were used to train the panelists. Panelists evaluated the samples using a “hybrid” descriptive analysis method (Grosso and Resurreccion, 2002) consisting of the Quantitative Descriptive Analysis (Tragon Corp., Redwood City, Calif., U.S.A.) and the Spectrum<sup>TM</sup> Descriptive Analysis (Sensory Spectrum, Inc., Chatham, N.J., U.S.A.) methods.

Panelists were then subjected to preliminary acuity tests to investigate their ability to recognize basic taste in solution, basic aromas, and to describe basic attributes related to Mafai Jeen fruit candy products. After the screening process eight panelists were selected. Five panelists were female and three were male in the range of 22-45 years old of age. Training consisted of: (1) Initial orientation session where panelists received detailed explanation about the descriptive sensory methodology and general description of the Mafai Jeen fruit candy product, (2) group meetings for lexicon development and selection of reference standards, and (3) individual training on the developed lexicon. During opening sessions panelists took part in a lexicon-generation exercise. They were provided with the Mafai Jeen fruit candy samples. They were asked to smell and taste them and list as many aromas and flavor terms as possible for each sample and discuss individual results to come up with a consensus. [Appendix Figure C 5](#) illustrates the panel during a group training

session. They were seated in a conference-type round table to facilitate communication. Throughout subsequent sessions panelists were exposed to different reference samples and they practiced the lexicon development process. During the last sessions panelists checked the developed lexicon and reduced the number of terms by eliminating redundant ones or those for which panelists could not reach consensus, agreed on precise definitions of the terms, and selected standards needed to describe them. After the final session, panelists had agreed on a list of twenty clearly defined terms (Table 3), the appropriate reference standards and their intensities on the 15-cm intensity line scale (Table 4).

Panelists attained individual training on the different intensities of the developed lexicon using a 15-cm structured line scale. They worked in partitioned booths, with positive airflow, free from distracting noises and odors. They were provided with room-temperature drinking water, unsalted crackers and an expectoration cup to cleanse their palate.

#### c. Product Evaluation

The eight panelists evaluated three samples Mafai Jeen fruit candy ( see appendix Figure C 6 ) using the developed lexicon, samples were placed in 6 x 8 cm zip locked plastic sachet; two pieces of candy were served for each sample. The panelists evaluated three samples once during a 60-minutes session and the evaluation was repeated two more times. Appendix Figure C 7 shows one of the trained panelists performing the evaluation. Panelists worked in partitioned booths. The panelists were instructed not to swallow the samples, and were asked to rinse their palate well with water between samples. For this purpose, panelists used room temperature drinking water and unsalted crackers. Subjects recorded the intensities of the attributes on the 15-cm structured line scale.

#### d. Data Analysis

Table 3 Attribute rated by the panel and their definitions used in the descriptive analysis of Mafai Jeen fruit candy.

Attribute	Definition
<b>Appearance</b>	
Glossy	Having a smooth, shiny, lustrous surface of sample
<b>Odor/Aroma</b>	
Pickled lemon	The aroma associated with pickled lemon
Dried preserved mandarin peel	The aroma associated with dried preserved mandarin peel
Pungent	Irritation, burnt and/or penetrating sensation in the interior of the nasal cavity
Caramel	Sweet aromatic associated with characteristic of over heat brown sugar
<b>Tastes</b>	
Sweet	The taste on the tongue associated with aqueous solution of sugar
Sour	The taste on the tongue associated with aqueous solution of acids
Bitter	The taste on the tongue associated with bitter agents such as caffeine or quinine
Astringent	A drying, puckering or tingling sensation on the surface and/or edges of tongue and mouth
<b>Flavor</b>	
pickled lemon	The flavor associated with pickled lemon
Dried preserved mandarin peel	The flavor associated with dried preserved mandarin peel
<b>Texture</b>	
<i>First bite</i>	
Hardness	The force required to compress the sample between the tongue and palate; compress through sample with tongue and palate

(Continued)

Table 3 (Continued)

Attribute	Definition
<i>Masticatory</i>	
Cohesiveness	Amount of deformation undergone by the material before rupture when biting completely through sample using molars; by place sample between molar teeth, compress and evaluate the amount of deformation before rupture
Crumble	The force required to break sample into small fragments or particles with molars
Sandy	A rough to the touch; chew sample 5 times and evaluate
Chewiness	Place sample in mouth and masticate at one chew per two second and at a force equal; evaluate the number of chews required to reduce sample to a state ready for swallowing
Roughness	Degree of abrasiveness of the product's surface as perceived by the tongue; chew sample with molars 8 times and evaluate
<i>Residual</i>	
Bitter	The residual taste on the tongue associated with bitter agents such as caffeine or quinine
Astringent	The residual of drying, puckering or tingling sensation on the surface and/or edges of tongue and mouth
Toothpack	The amount of product packed into the crowns of your teeth after mastication; chew sample up to 8 times, expectorate and feel the surface of the crowns of the teeth with tongue

**Table 4** Standard reference intensity ratings used in descriptive tests for Mafai Jeen fruit candy.

Attribute	Reference	Intensity <sup>1</sup> (cm)
<b>Appearance</b>		
Glossy	Warm-up sample <sup>2</sup>	5
	Ka la mae, Savei (Jiraporn, Chiangmai, Thailand)	15
<b>Odor/Aroma</b>		
pickled lemon	Warm-up sample <sup>2</sup>	3
	Pickled lemon 1 cm cube	15
Dried preserved mandarin peel	Warm-up sample <sup>2</sup>	2
	Dried preserved mandarin peel (Jiabao brand, Guangdong Jiabao group Co., Ltd, Guangdong)	15
Pungent	Warm-up sample <sup>2</sup>	5
	Dried Mafai Jeen fruit	10
	3 drop of white vinegar (Thai Q.P. Co., Ltd., Rajburi, Thailand)	15
Caramel	Warm-up sample <sup>2</sup>	0
	Werther's Original chewy toffees (August Storck KG, Berlin)	3
<b>Tastes</b>		
Sweet	Warm-up sample <sup>2</sup>	12
	10.0% sucrose in deionized water	10
	15.0% sucrose in deionized water	15
Sour	Warm-up sample <sup>2</sup>	10
	0.08% citric acid in deionized water	5
Bitter	Warm-up sample <sup>2</sup>	4
	0.05% caffeine in deionized water	12
Astringent	Warm-up sample <sup>2</sup>	3
	0.1% alum in deionized water	5
	Dried preserved mandarin peel (Jiabao brand, Guangdong)	6

(Continued)

Table 4 (Continued)

Attribute	Reference	Intensity <sup>1</sup> (cm)
	Jiabao group Co., Ltd, Guangdong)	
	0.15% alum in deionized water	15
Flavor		
pickled lemon	Warm-up sample <sup>2</sup>	5
	Pickled lemon 1 cm cube	15
Dried preserved	Warm-up sample <sup>2</sup>	2
mandarin peel	Dried preserved mandarin peel (Jiabao brand, Guangdong Jiabao group Co., Ltd, Guangdong)	12
Texture		
<i>First bite</i>		
Hardness	Warm-up sample <sup>2</sup>	3
	Marmalade (Best Foods, Malee Bangkok Co., Ltd., Samtuprakarn, Thailand)	2
	5 min boiled egg white	5
	Mint flavor candy (MYMINT brand, Boonprasert conf. Samutsongkharm, Thailand)	13
<i>Masticatory</i>		
Cohesiveness	Warm-up sample <sup>2</sup>	4
	PIPO JELLY, EURO, Pipo brand, variety flavored jelly dessert	2
	Mint flavor candy (MYMINT brand, Boonprasert conf. Samutsongkharm, Thailand)	13
	SUGUS (SUGUS chewy candy, Kraft Jacobs Ltd., Switz.)	15
Crumble	Warm-up sample <sup>2</sup>	1
	Ka nom phing (Cookie Kaset, Institute of Food Research and Prod. Dev. (IFRPD), KU, Bangkok, Thailand)	7
	Coconut sugar cube	12

(Continued)

Table 4 (Continued)

Attribute	Reference	Intensity <sup>1</sup> (cm)
	Tob tub peanut candy ( Taveephol product Co., Ltd, Talingchan, Bangkok, Thailand)	15
Sandy	Warm-up sample <sup>2</sup>	3
	Coconut sugar cube	7
	Ka nom phing (Cookie Kaset, Institute of Food Research and Prod. Dev. (IFRPD), KU, Bangkok, Thailand)	10
Chewiness	Warm-up sample <sup>2</sup>	6
	Banana candy (Kleaw Jai, Maeban Nongkasamakkee group, Bungkum, Bangkok, Thailand)	13
Roughness	Warm-up sample <sup>2</sup>	4
	PIPO JELLY, EURO, Pipo brand, variety flavored jelly dessert	0
	Hawthorn piece (Guan Du Qu Qian Wei Ying Xia, 157 Kun Ming, Yun Nan)	6
<i>Residual</i>		
Bitter	Warm-up sample <sup>2</sup>	4
	0.05% caffeine in deionized water	12
Astringent	Warm-up sample <sup>2</sup>	2
	0.1% alum in deionized water	5
	Dried preserved mandarin peel (Jiabao brand, Guangdong Jiabao group Co., Ltd, Guangdong)	6
	0.15% alum in deionized water	15
Toothpack	Warm-up sample <sup>2</sup>	2
	Banana candy (Kleaw Jai, Maeban Nongkasamakkee group, Bungkum, Bangkok, Thailand)	10

<sup>1</sup>Intensity rating are based on 15-cm structured line scale

<sup>2</sup>Warm-up sample consist of 59% Mafai Jeen puree, 35% sugar, 5% glucose syrup, and 1% pectin and processing step as followed by figure 3.

The data were analyzed using univariate statistical analysis. Analysis of Variance (ANOVA, SPSS version 10.0.1, 2001) was performed to determine significant effects of the attribute intensities in each of the three Mafai Jeen fruit candy.

#### 4.3.5 Consumer Test

Consumers (n = 135) were randomly recruited from Kasetsart University and from a local public area in Bangkok. The criteria for recruitment were: at least 18 years old, not allergic to fruit candy products, and positive attitude. Consumers were instructed on the procedure to be followed. They were informed that each sample was randomly coded with a 3-digit number. These numbers corresponded to those appearing on each of the three pages of the questionnaire. Prior to the product evaluation, participants were asked to complete a demographic and socioeconomic survey, which included questions regarding age, gender, education level, employment status, and household income.

Three samples of Mafai Jeen candy piece added 2.0% maltodextrin, 1.5% pectin, and 1.0% CMC were tasted at room temperature in a random 3-digit number coded in a sequential monadic presentation. Room-temperature drinking water and unsalted crackers were provided to consumers to cleanse their palate between samples evaluation, in order to minimize sensory carryover and/or fatigue effects. Consumers were told to chew at least half of the piece and to evaluate the three samples for acceptability of overall appearance, texture, taste, sweetness and overall liking using a 9-point hedonic scale (1=dislike extremely, 5= neither like, nor dislike, and 9=like extremely) (Peryam and Pilgrim, 1957). Consumers were also asked to rate the sweetness of each Mafai Jeen fruit candy (added 2.0% maltodextrin one, added 1.5% pectin one, and added 1.0% CMC one) on a 3-point “just about right” (JAR) scale with “just about right”= 2, “too weak”=1, and “too strong”=3. Participants evaluated acceptance of the product using a yes/no scale (Moscowitz, 1994). Finally, consumers evaluated purchase intent

(yes/no) and purchase intent after additional information about health benefits of the Mafai Jeen fruit candy had been provided.

Data were analyzed using univariate statistical analyses. A Randomized Block Design was followed and Analysis of Variance (ANOVA, SPSS version 10.0.1) was performed to determine differences in acceptability for each of the sensory attributes among the three samples. The Duncan post-hoc test was applied to accomplish all paired-wise comparisons of acceptability of each of the three Mafai Jeen fruit candy.

McNemar test was performed to analyze the change in probability of purchase intent before and after consumers had been informed about the health benefits of the product. The McNemar's test represents a comparison of dependent proportions for binary response variables. It is a two-related sample difference test, that follows a Chi-square distribution with 1 df (Agresti, 1996). Consumers are categorized in two categories, in a "before" condition and then the same consumers are re-categorized in an "after" condition (O'Mahony, 1986). The null hypothesis ( $H_0: \pi_{+1} - \pi_{1+} = 0$  or  $\pi_{21} - \pi_{12} = 0$ ) stated that there was no significant difference in the probability of buying the product before and after consumers had been informed about its health benefits. In other words, the question was whether the differences between the probability of those who answered yes after ( $\pi_{+1}$ ) and the probability of those who answered yes before ( $\pi_{1+}$ ) is significant, or whether it was merely chance. The aim is to know if participants were influenced or not by the fact that they were informed about health benefits of the product, and therefore their opinions changed from a "before" status to an "after" status. To complement this test and obtain more detailed understanding, a 95% confidence interval for the difference of proportions was calculated. The difference of two sample marginal proportions ( $p_{+1} - p_{1+}$ ) estimates the true difference ( $\pi_{+1} - \pi_{1+}$  or  $\pi_{21} - \pi_{12}$ ). Equation (7) indicates the formula used to calculate sample proportions, where  $n_{ij}$  is the number of subjects making response  $i$  at the first question (before), and response  $j$  at the second question (after knowing that the product contained health promoting ingredients) and  $N$  is the

total number of responses. Equation (8) is used to calculate confidence interval of difference of proportions. The term  $(p_{+1} - p_{1+})$  indicates the difference between the proportion of participants that answered 1 (yes) after knowing that the product contained health benefits ( $p_{+1}$ ), and the proportion of participants that answered 1 (yes) before knowing that the product contained health benefits ( $p_{1+}$ ). Equation (8) is useful to calculate 95% confidence interval of such difference. The term  $z_{\alpha/2}$  denotes the standard normal percentile having a right-tail probability equal to  $\alpha/2$  (in this case a 95% interval,  $\alpha=0.05$ ,  $z_{\alpha/2} = 1.96$ ). ASE is the estimated standard error for the proportion difference and was calculated using equation (9); where  $p_{11}$ = proportion of subjects that answered 1 (yes) before knowing and 1(yes) after knowing,  $p_{22}$ = proportion of subjects that answered 2(no) before knowing and 2(no) after knowing that the product contained health promoting minerals and vitamins. A 95% confidence interval denotes that the calculated difference of proportions is 95% of the time correct, with an alpha level set at 0.05, which correspond to the 5% error allowed. When 0 is included in the confidence interval, then there is no significant difference.

$$p_{ij} = n_{ij} / N \dots\dots\dots(7)$$

$$(p_{+1} - p_{1+}) \pm z_{\alpha/2} (ASE) \dots\dots\dots(8)$$

$$ASE = [ p_{1+} x(1- p_{1+}) + p_{+1} x(1- p_{+1}) - 2x(p_{11}p_{22} - p_{12}p_{21}) / N ]^{1/2} \dots\dots\dots(9)$$

#### 4.4 To Study on Shelf Life of Developed Product

The Mafai Jeen fruit candy were cut into 0.5 x 0.5 x 0.5 inch and individual wrapped using oriented polypropylene (OPP) film 25  $\mu$ m thickness.

To consider the economics of the packaging materials and also the product deterioration characteristics, pouches made of the following materials were selected for shelf life studies; 30  $\mu$ m OPP film and aluminum laminated foil pouches (Strongpack Public Co., Ltd., Bangkok). A total of 20 candy were packed ( $\approx 70$

g/bag) in two different types of packaging material with dimensions of 18 cm x 13 cm., hermetically heat sealed on the edges and stored at 25 °C and 40 °C. The Mafai Jeen fruit candy samples were withdrawn periodically in order to monitor;

#### 4.4.1 Moisture Content

Moisture content of the product was measured using the methods of Association of Official Analytical Chemists (AOAC, 1990).

#### 4.4.2 Water Activity ( $a_w$ )

The water activity of Mafai Jeen fruit candy was determined at 25 °C using Thermoconstanter (Novasina, MLK, Switzerland), equipped with a temperature-controlled system which allow to have a temperature stable sampling environment. The equipment was calibrated with saturated salt solution in the  $a_w$  range of 0.43 – 0.75 (Favetto *et al.*, 1983). For each determination, five replication were obtained and the average reported; under these conditions reliability of this meter is about  $\pm 0.003 a_w$ .

#### 4.4.3 Color Assessment

The color of Mafai Jeen fruit candy was measured with a tristimulus reflectance colorimeter using a spectrophotometer (CM 3500d, Minolta Camera Co., Ltd, Tokyo, Japan). Three replicates of 10 pieces were used for each storage time sample. Color is recorded using a CIE –  $L^*$   $a^*$   $b^*$  uniform color space, where  $L^*$  indicates lightness on 0 (black) to 100 (white),  $a^*$  indicates chromaticity on a green ( - ) to red ( + ) axis, and  $b^*$  chromaticity on a blue ( - ) to yellow ( + ) axis.

#### 4.4.4 Sensory Analysis

To obtain consumer acceptance of Mafai Jeen fruit candy stored at different temperatures and time, fifty untrained consumers, consisting of employees,

students and faculty members, were recruited from the Kasetsart University for the consumer test. Panelists were recruited if (a) they are not allergic to fruit candy, (b) their ages are between 19 and 65 years, and (c) they eat fruit candy products at least once a month. Consumers who participated in one of the consumer tests did not necessarily participate in another test. Consumers recorded their answers on paper ballots in terms of overall acceptance, as well as acceptance of color, taste, and texture, using a 9-point hedonic scale, with 1 = dislike extremely, 5 = neither like nor dislike, and 9 = like extremely (Peryam, 1964).

## RESULTS AND DISCUSSION

### 1. To Identify the Aroma Compounds of Mafai Jeen

#### 1.1 The Aroma Compounds of Fruits, Seeds, and Leaves

In order to detect the “true” fragrance composition experienced by the consumers, a headspace sampling technique was performed in this experiment. Over 72 compounds were isolated and over 60 characterized from their retention index, mass spectra and data from the literature. The aroma compounds of leaves, flesh, skin of fruit and seeds are summarized in [Table 5](#), which shows that these compounds were found in different percentages in the various parts of the plant. The majority of these components were found to belong to the hydrocarbon fraction, with percentages ranging from 50% in the leaves, 77% in the flesh, to 96% in the skin and 99% in the seeds. Among the components of the hydrocarbon fraction, the predominant compounds were found to be sesquiterpenes in the leaves, and monoterpenes in the flesh, skin and seeds.

A total of 39 components were identified in the headspace of leaves, amounting to 86% of the total volatiles. The sample was dominated by the sesquiterpenes (28%), with  $\beta$ -bisabolene,  $\beta$ -caryophyllene, and  $\alpha$ -zingiberene as the main components. Monoterpenes were fewer (22%) with sabinene (15%) as the main component. In addition to the hydrocarbons, an ester, 3-hexenyl 2-methylbutanoate (0.19%), along with its alcohol, 3-hexen-1-ol (0.17%) was characterized in the headspace of leaves. The compounds 3-hexen-1-ol and its acetate were often found in green leaves (Hatanaka, 1993). Sesquiterpenes,  $\beta$ -caryophyllene (7.72%) and humulene (0.39%), and an ester (3-hexenyl 2-methylbutanoate, 0.19%) were released in response to the attack by the insect of *Spodoptera* in cotton plantlets (Loughrin *et al.*, 1994). Camciuc *et al.* (1998) proposed that the biological activity of some of these compounds seems to support the hypothesis of their role in defence against insects. The monoterpenes and esters present in Mafai Jeen leaves may act as solvents and

also have a synergic action with molecules having irritant properties. Interestingly, significant amount of *ar*-curcumene (1.27%),  $\alpha$ -zingiberene (6.52%) and  $\beta$ -bisabolene (9.88%) were characterized in the headspace of leaves as well. Rani (1999) reported that the essential oil from the rhizomes of *Zingiber officinale* Roacoe contains *ar*-curcumene (20%),  $\alpha$ -zingiberene (22%) and  $\beta$ -bisabolene (14%). He postulated that bisabolyyl cation may be derived from farnesylpyrophosphate. Bisabolyyl cation is the penultimate precursor of *ar*-curcumene,  $\alpha$ -zingiberene and  $\beta$ -bisabolene; and two 1,2-hydrogen shifts lead to the formation of  $\alpha$ -zingiberene whereas one 1,2-hydrogen shift leads to the formation of *ar*-curcumene. The chemical composition of the essential oil obtained from the rhizomes of *Z. officinale* Roscoe was characterized by the presence of *ar*-curcumene (22.1%), zingiberene (11.7%), beta-bisabolene (11.2%) and cadina-1,4-diene (12.5%). It is reasonable that in Sri Lanka (Kumar *et al.*, 1995) the leaves are used as a substitute for curry leaf in cooking.

Monoterpenes constituted the major part of the fragrance emitted from Mafai Jeen flesh, skin and seed. The monoterpene hydrocarbon fraction (76%) dominated the flesh, with sabinene (50.6%) and 1,4-cyclohexadiene (6.2%) as the main components. The alcohol fraction represented 17.5% of the total volatiles with 3-cyclohexen-1-ol (15%) as the major component.

In the skins, 30 components could be identified, amounting to 96.7% of the total volatiles. The sample was dominated by the monoterpenes (94%), with sabinene (69%),  $\alpha$ -phellandrene (10.6%), and  $\alpha$ -pinene (9.4%) as the main components.

In the seeds, 26 components could be identified, amounting to 99% of the total volatiles. The sample was dominated by the monoterpenes (98%), with sabinene (84%),  $\alpha$ -pinene (4.3%),  $\alpha$ -phellandrene (3.1%), and myrcene (2.9%) as the main components.

**Table 5** Volatile compounds identified in Mafai Jeen (leaf, flesh, skin of fruit, and seed) using headspace sampler with HP-5MS non-polar column.

No	Compounds	RI	% Relative peak area				ID
			Leaf	Flesh	Skin	Seed	
<i>Monoterpene hydrocarbons</i>							
1.	tricyclene	928	<i>t</i>	<i>t</i>	0.03	<i>t</i>	MS, RI <sub>1</sub>
2.	$\alpha$ -thujene	931	<i>t</i>	<i>t</i>	0.02	0.59	MS, RI <sub>1</sub>
3.	$\alpha$ -pinene	939	1.99	2.08	9.41	4.26	MS, RI <sub>1</sub>
4.	camphene	945	0.98	<i>t</i>	0.47	0.04	MS, RI <sub>1</sub>
5.	$\beta$ -pinene	967	<i>t</i>	0.21	0.17	<i>t</i>	MS, RI <sub>1</sub>
6.	limonene	1026	<i>t</i>	0.21	<i>t</i>	<i>t</i>	MS, RI <sub>1</sub>
7.	myrcene	993	1.10	1.70	3.15	2.94	MS, RI <sub>1</sub>
8.	$\alpha$ -phellandrene	1001	1.38	5.03	10.63	3.08	MS, RI <sub>1</sub>
9.	3-carene	1010	<i>t</i>	<i>t</i>	0.10	<i>t</i>	MS, RI <sub>1</sub>
10.	(+) 4-carene	1018	<i>t</i>	3.98	0.40	1.13	MS, RI <sub>1</sub>
11.	sabinene	973	14.92	50.64	69.07	83.56	MS, RI <sub>1</sub>
12.	trans- $\beta$ -ocimene	1035	<i>t</i>	<i>t</i>	<i>t</i>	0.02	MS, RI <sub>1</sub>
13.	1,3,6-octatriene	1039	1.96	<i>t</i>	0.06	<i>t</i>	MS, RI <sub>1</sub>
14.	1,4-cyclohexadiene	1043	<i>t</i>	6.19	0.32	<i>t</i>	MS, RI <sub>1</sub>
15.	$\gamma$ -terpinene	1057	<i>t</i>	<i>t</i>	0.04	1.95	MS, RI <sub>1</sub>
16.	cyclohexene	1065	<i>t</i>	6.50	0.17	0.39	MS, RI <sub>1</sub>
	total		<u>22.34</u>	<u>76.54</u>	<u>94.05</u>	<u>97.96</u>	
<i>Sesquiterpene hydrocarbons</i>							
1.	copaene	1373	0.28	<i>t</i>	<i>t</i>	<i>t</i>	MS, RI <sub>1</sub>
2.	$\beta$ -caryophyllene	1417	7.72	<i>t</i>	<i>t</i>	0.55	MS, RI <sub>1</sub>
3.	$\alpha$ -bergamotene	1427	0.71	<i>t</i>	0.20	0.03	MS, RI <sub>1</sub>
4.	(+)-aromadendrene	1436	0.08	<i>t</i>	<i>t</i>	<i>t</i>	MS, RI <sub>1</sub>
5.	isosativene	1441	0.38	<i>t</i>	0.07	0.01	MS, RI <sub>1</sub>
6.	$\beta$ -santalene	1444	<i>t</i>	<i>t</i>	0.02	<i>t</i>	MS, RI <sub>1</sub>
7.	$\alpha$ -humulene	1447	0.39	<i>t</i>	0.02	0.03	MS, RI <sub>1</sub>

(Continued)

Table 5 (Continued)

No	Compounds	RI	% Relative peak area				ID
			Leaf	Flesh	Skin	Seed	
8.	ar-curcumene	1475	1.27	0.12	0.87	0.03	MS, RI <sub>1</sub>
9.	allaromadendrene	1478	<i>t</i>	<i>t</i>	0.10	<i>t</i>	MS, RI <sub>1</sub>
10.	$\alpha$ -zingiberene	1486	6.52	<i>t</i>	<i>t</i>	0.06	MS, RI <sub>1</sub>
11.	bicyclogermacrene	1490	0.37	<i>t</i>	<i>t</i>	0.01	MS, RI <sub>1</sub>
12.	$\alpha$ -farnesene	1494	<i>t</i>	<i>t</i>	0.95	<i>t</i>	MS, RI <sub>1</sub>
13.	$\beta$ -bisabolene	1496	9.88	<i>t</i>	<i>t</i>	0.15	MS, RI <sub>1</sub>
14.	$\beta$ -sesquiphellandrene	1512	0.70	<i>t</i>	0.30	<i>t</i>	MS, RI <sub>1</sub>
15.	$\delta$ -cadinene	1524	0.33	<i>t</i>	<i>t</i>	<i>t</i>	MS, RI <sub>1</sub>
	total		<u>27.69</u>	<u>0.12</u>	<u>2.22</u>	<u>0.85</u>	
<i>Alcohols</i>							
1.	ethanol		<i>t</i>	2.46	<i>t</i>	<i>t</i>	MS
2.	1-penten-3-ol		1.89	<i>t</i>	<i>t</i>	<i>t</i>	MS
3.	cis 2-pentenol		0.71	<i>t</i>	<i>t</i>	<i>t</i>	MS
4.	3-hexen-1-ol	857	0.17	<i>t</i>	<i>t</i>	<i>t</i>	MS, RI <sub>1</sub>
5.	linalool	1086	2.25	<i>t</i>	0.16	<i>t</i>	MS, RI <sub>1</sub>
6.	3-cyclohexen-1-ol	1097	<i>t</i>	15.07	0.28	0.51	MS, RI <sub>1</sub>
7.	3-cyclohexen-1-methanol	1106	<i>t</i>	<i>t</i>	0.02	<i>t</i>	MS, RI <sub>1</sub>
8.	$\beta$ -fenchyl alcohol	1109	<i>t</i>	0.54	<i>t</i>	0.06	MS, RI <sub>1</sub>
	total		<u>5.02</u>	<u>18.06</u>	<u>0.46</u>	<u>0.57</u>	
<i>Aldehydes</i>							
1.	propanal		1.63	<i>t</i>	<i>t</i>	<i>t</i>	MS
2.	butanal		8.61	<i>t</i>	<i>t</i>	0.02	MS
3.	hexanal	802	1.55	0.47	0.04	<i>t</i>	MS, RI <sub>1</sub>
4.	2-hexenal	854	1.46	<i>t</i>	<i>t</i>	<i>t</i>	MS, RI <sub>1</sub>
5.	benzaldehyde	958	2.56	<i>t</i>	<i>t</i>	0.02	MS, RI <sub>1</sub>
6.	benzeneacetaldehyde	1037	0.30	<i>t</i>	<i>t</i>	<i>t</i>	MS, RI <sub>1</sub>
	total		<u>16.12</u>	<u>0.47</u>	<u>0.04</u>	<u>0.04</u>	

(Continued)

Table 5 (continued)

No	Compounds	RI	% Relative peak area				ID
			Leaf	Flesh	Skin	Seed	
<i>Esters</i>							
1.	bornyl acetate	1286	<i>t</i>	<i>t</i>	<i>t</i>	0.01	MS, RI <sub>1</sub>
2.	cis-3-hexenyl 2-methylbutanoate	1218	0.19	<i>t</i>	0.01	<i>t</i>	MS, RI <sub>1</sub>
3.	geranyl acetate	1357	<i>t</i>	<i>t</i>	0.02	<i>t</i>	MS, RI <sub>1</sub>
	total		<u>0.19</u>	<u>0.00</u>	<u>0.03</u>	<u>0.01</u>	
<i>Ketones</i>							
1.	2-propanone		3.02	<i>t</i>	<i>t</i>	<i>t</i>	MS, RI <sub>1</sub>
2.	ethanone		0.20	<i>t</i>	<i>t</i>	<i>t</i>	MS, RI <sub>1</sub>
3.	6-methyl-5-hepten-2-one	976	2.26	<i>t</i>	<i>t</i>	<i>t</i>	MS, RI <sub>1</sub>
4.	2-nonanone	1079	3.42	<i>t</i>	<i>t</i>	0.01	MS, RI <sub>1</sub>
5.	2-cyclohexen-1-one	1099	<i>t</i>	<i>t</i>	0.03	0.01	MS, RI <sub>1</sub>
	total		<u>8.90</u>	<u>0.00</u>	<u>0.03</u>	<u>0.02</u>	
<i>Heterocyclics</i>							
1.	2-methylfuran		1.10	<i>t</i>	<i>t</i>	<i>t</i>	MS, RI <sub>1</sub>
2.	2-ethylfuran		4.61	<i>t</i>	<i>t</i>	<i>t</i>	MS, RI <sub>1</sub>
	total		<u>5.72</u>	<u>0.00</u>	<u>0.00</u>	<u>0.00</u>	
<i>Carboxylic acids</i>							
1.	acetic acid		0.94	2.65	0.08	0.03	MS, RI <sub>1</sub>
2.	benzoic acid	1163	0.16	<i>t</i>	<i>t</i>	<i>t</i>	MS, RI <sub>1</sub>
	total		<u>1.10</u>	<u>2.65</u>	<u>0.17</u>	<u>0.03</u>	
<i>Hydrocarbons</i>							
1.	styrene	921	0.13	<i>t</i>	<i>t</i>	<i>t</i>	MS, RI <sub>1</sub>
2.	(E)-4,8-dimethyl-1,3,7-nonatriene	1089	1.22	<i>t</i>	<i>t</i>	<i>t</i>	MS, RI <sub>1</sub>
3.	3-methyl-4-brendene	1095		0.13	<i>t</i>	<i>t</i>	MS, RI <sub>1</sub>
	total		<u>1.35</u>	<u>0.13</u>	<u>0.00</u>	<u>0.00</u>	
	Total		86.18	97.44	96.73	99.42	
60	n		39	16	30	26	

RI = programmed temperature retention indices relative to the homologous series of n-alkanes (C5-C25); RI<sub>1</sub> = retention data in literature; *t* = traces < 0.01%; ID = identification method

It should be noted that 1-phellandrene was abundant in all of the samples examined,  $\alpha$ -pinene was more abundant in skin than in seed and flesh, whereas 3-cyclohexen-1-ol was distinctive component in the flesh. Sulfur-containing compounds could not be identified in any part of the samples examined. The composition of the volatile compounds of the leaves and seeds of *C. lansium* was distinctly different from that previously studied from Hainan Island, China (Zhao *et al.*, 2004) by the absence of the  $\beta$ -santalol, bisabolol and ledol that represents the major volatile component in the leaves and seeds. The steam distillation procedure was applied in their experiment to obtain the essential oils from the flowers, leaves, sarcocarps and seeds of *C. lansium*, respectively. High boiling point alcohols, such as  $\beta$ -santalol (35.2%), bisabolol (13.7%) and ledol (6.5%) in leaves, were extracted under much higher temperature (boiling point of water) compared to the extraction conditions in this experiment (80 °C).

Sabinene (15%, 51%, 69% and 84% in leaf, flesh, skin and seed, respectively) was the major headspace volatile in Mafai Jeen. Sabinene was found to be extremely abundant in the plant and the fresh fruit essential oil of *Peucedanum verticillare* (Fraternale *et al.*, 2000). The emission of sabinene and limonene (trace amounts; see Table 5) was two to three times higher in the middle of the light cycle than it was in darkness in flowers and leaves of *Brassica napus* in situ (Jackobsen *et al.*, 1972). The essential oils from the leaves of *Clausena anisata* (Willd.) J.D. Hook ex Benth, were isolated and found to contain mainly sabinene (33.0%), germacrene-D (17.0%), Z- $\beta$ -ocimene (6.0%), germacrene-B (5.5%), (E)- $\beta$ -ocimene (4.9%) and terpinen-4-ol (4.7%) (Gundidza *et al.*, 1994).

The headspace composition of Mafai Jeen samples gives a better overall representation of the compounds detected by smell as compared with that of the steam distillate. The study successfully isolated and characterized the low-temperature volatile aromatic compounds in Mafai Jeen using the headspace sampling technique.

## 1.2 Aroma Compounds of Mafai Jeen Fruits by HS-SPME Analysis

The analysis of aroma compounds in Mafai Jeen fruits through solid phase microextraction has not been previously reported. However, the selection of an appropriate fiber depends on the compounds and the food to be analyzed. In this study, three types of coating were chosen: (1) DVB/CAR/PDMS, (2) PA, and (3) PDMS. Identified volatile compounds with their relative peak area are summarized in [Table 6](#). In total, 88 compounds were detected in the Mafai Jeen aroma. The three fibers gave results that differed quantitatively with 27, 44, and 24 compounds absent from DVB/CAR/PDMS, PA and PDMS, respectively. Therefore using DVB/CAR/PDMS or PDMS fibers will give more information on the aroma of Mafai Jeen than PA fiber.

Inspection of [Table 6](#) shows that the chemical classes found were: 15 monoterpene hydrocarbons, 23 sesquiterpene hydrocarbons, 16 alcohols, 5 aldehydes, 10 esters, 6 ketones, 1 sulfur-containing compound, 8 carboxylic acids, and 4 hydrocarbons. The most abundant compound was sabinene (22.5-37.2%). Other major constituents were  $\alpha$ -farnesene, isosativene, and trans- $\beta$ -farnesene. Twenty six of these compounds were found in all extracts. There were, 1S- $\alpha$ -pinene, myrcene,  $\alpha$ -phellandrene, sabinene, isosativene,  $\alpha$ -bergamotene, 1,4,7-cycloundecatriene, trans- $\beta$ -farnesene,  $\gamma$ -curcumene,  $\alpha$ -farnesene, 1H-3a,7-methanoazulene, trans- $\gamma$ -bisabolene, linalool L, 2-cyclohexen-1-ol, 3-cyclohexen-1-ol, terpinene-3-ol, nerolidol, 5-isocedranol, cis- $\alpha$ -santalol, santalol, hexanal,  $\alpha$ -sinensal, geranyl acetate, ethylic acid, acetic acid, and 2,6-octadienoic acid. Only some compounds were detected by PA; those were  $\alpha$ -terpinene, terpinolene,  $\beta$ -santalene, trans-carvyl acetate, 2H-benzimidazol-2-one, methanone, and pentadecanoic acid. DVB/CAR/PDMS and PDMS fibers showed similarity in aroma compounds detected, i.e., camphene,  $\beta$ -pinene, (+)-4-carene, 1,3,6-octatriene, zingiberene, cis- $\alpha$ -bisabolene,  $\alpha$ -terpineol, 2-cyclohexen-1-ol,  $\beta$ -santalol, 2-hexanal, cis-3-hexenyl 2-methylbutanoate, sabinyl acetate, neryl acetate, 2-nonanone, piperitone isomer, and disulfide.

**Table 6** Volatile aroma compounds identified in the Mafai Jeen fruit in order of their retention time ( $t_R$ ) using SPME with 100  $\mu\text{m}$  polydimethylsiloxane (PDMS), 85  $\mu\text{m}$  polyacrylate (PA), and 50/30  $\mu\text{m}$  divinylbenzene/carboxen/PDMS (DVB/CAR/PDMS) fibers coupled to GC–MS.

No.	Compounds	MW	$t_R$ (Min)	% Relative peak area		
				DVB/CAR/PDMS	PA	PDMS
<i>Monoterpene hydrocarbons</i>						
1	$\alpha$ -thujene	136	8.649	0.03	-	-
2	$\alpha$ -pinene	136	8.895	0.90	0.59	1.91
3	camphene	136	9.457	0.06	-	0.09
4	$\beta$ -pinene	136	10.611	0.03	-	0.06
5	myrcene	136	11.328	1.51	0.60	1.28
6	$\alpha$ -phellandrene	136	11.871	4.39	2.10	4.36
7	$\delta$ -3-carene	136	12.083	-	-	0.04
8	$\alpha$ -terpinene	136	12.362	-	0.08	-
9	(+)-4-carene	136	12.386	0.52	-	0.31
10	sabinene	136	13.170	32.78	22.50	37.20
11	1,3,6-octatriene	136	13.867	0.10	-	0.04
12	1,4-cyclohexadiene	136	14.276	-	0.20	0.31
13	$\gamma$ -terpinene	136	14.305	0.43	-	-
14	cyclohexene	136	15.632	0.25	-	-
15	terpinolene	136	15.632	-	0.10	-
	total			<u>41.00</u>	<u>26.16</u>	<u>45.61</u>
	n			<u>11</u>	<u>7</u>	<u>10</u>
<i>Sesquiterpene hydrocarbons</i>						
1	$\delta$ -elemene	204	26.250	0.02	-	-
2	zingiberene	204	27.520	0.64	-	0.09
3	isosativene	204	28.424	7.65	4.12	8.43
4	$\alpha$ -bergamotene	204	28.828	2.03	1.59	2.23

(Continued)

Table 6 (Continued)

No.	Compounds	MW	$t_R$ (Min)	% Relative peak area		
				DVB/CAR/PDMS	PA	PDMS
5	$\beta$ -sesquiphellandrene	204	29.025	0.64	0.55	-
6	$\beta$ -santalene	204	29.250	-	0.27	-
7	2-methyl-3-methylene	204	29.150	-	-	0.18
8	1,4,7-cycloundecatriene	204	29.318	0.38	0.28	0.33
9	trans- $\beta$ -farnesene	204	29.448	7.14	4.10	7.90
10	$\alpha$ -longipinene	204	29.684	-	-	0.06
11	$\gamma$ -curcumene	204	30.020	0.86	0.80	0.95
12	$\beta$ -cubebene	204	30.083	-	0.10	0.18
13	1,6,10-dodecatriene	204	30.165	-	-	0.09
14	$\alpha$ -farnesene	204	30.833	14.64	11.72	15.11
15	1H-3a,7-methanoazulene	204	30.972	1.59	1.81	0.15
16	trans- $\gamma$ -bisabolene	204	31.016	0.48	0.25	3.22
17	$\beta$ -himachalene	204	31.035	2.61	-	-
18	$\gamma$ -1-cadinene	204	31.194	-	-	0.11
19	cis- $\alpha$ -bisabolene	204	31.655	0.31	-	0.08
20	$\beta$ -bisabolene	204	31.665	-	1.08	0.47
21	$\gamma$ -elemene	204	32.809	-	-	0.05
22	$\alpha$ -cedrene	204	33.882	-	-	0.15
23	italicene	204	33.882	-	-	0.06
	total			<u>39.00</u>	<u>26.67</u>	<u>39.81</u>
	n			<u>14</u>	<u>12</u>	<u>20</u>
<i>Alcohols</i>						
1	ethanol	46	1.556	0.05	-	-
2	linalool L	154	16.281	1.78	0.76	0.51

(continued)

Table 6 (continued)

No	Compounds	MW	$t_R$ (Min)	% Relative peak area		
				DVB/CAR/PDMS	PA	PDMS
3	2-cyclohexen-1-ol	154	17.128	0.14	0.45	0.24
4	1-terpineol	154	17.969	0.12	0.16	-
5	3-cyclohexen-1-ol	154	19.729	3.10	1.10	0.84
6	$\alpha$ -terpineol	154	20.340	0.16	-	0.04
7	terpinene-3-ol	154	21.085	0.11	0.15	0.08
8	nerol	154	21.980	0.12	-	-
9	trans-geraniol	154	23.033	0.33	0.21	-
10	2-cyclohexen-1-ol	194	25.904	0.42	-	0.21
11	nerolidol	222	32.160	1.29	1.28	1.13
12	5-isocedranol	220	34.699	0.64	0.89	0.64
13	cis- $\alpha$ -santalol	220	35.324	0.58	0.85	0.80
14	$\beta$ -santalol	220	35.858	0.16	-	0.22
15	santalol	220	38.277	0.10	3.18	1.60
16	phenol	228	44.784	-	7.36	1.63
	total			<u>9.11</u>	<u>16.38</u>	<u>7.94</u>
	n			<u>15</u>	<u>11</u>	<u>12</u>
<i>Aldehydes</i>						
1	hexanal	100	4.682	0.18	0.06	0.03
2	2-hexanal	98	6.120	0.50	-	0.02
3	nonanal	142	16.440	0.11	-	-
4	decanal	156	19.104	-	-	0.03
5	$\alpha$ -sinensal	218	36.531	0.25	0.51	0.39
	total			<u>1.04</u>	<u>0.57</u>	<u>0.47</u>
	n			<u>4</u>	<u>2</u>	<u>4</u>
<i>Esters</i>						
1	cis-3-hexenyl iso-butyrate	170	18.296	0.07	-	-

(continued)

Table 6 (continued)

No.	Compounds	MW	$t_R$ (Min)	% Relative peak area		
				DVB/CAR/PDMS	PA	PDMS
2	cis-3-hexenyl 2-methylbutanoate	184	22.191	0.14	-	0.04
3	sabinyl acetate	194	25.740	0.31	-	0.10
4	trans-carvyl acetate	194	25.899	-	0.12	-
5	neryl acetate	196	26.745	0.61	-	0.28
6	geranyl acetate	196	27.327	1.31	0.34	0.64
7	cis-3-hexenyl benzoate	204	32.338	0.04	-	-
8	sesquisabinene hydrate	222	32.559	-	0.66	0.61
9	diethyl phthalate	222	32.891	-	0.89	0.12
10	(+)-methyl (E) $\alpha$ -santalenoate	248	37.440	-	0.09	0.11
	total			<u>2.49</u>	<u>2.10</u>	<u>1.89</u>
	n			<u>6</u>	<u>5</u>	<u>7</u>
<i>Ketones</i>						
1	ethyl vinyl ketone	84	2.739	0.02	-	-
2	2-nonanone	142	15.877	0.07	-	0.02
3	2-cyclohexen-1-one	138	20.128	0.15	-	-
4	piperitone isomer	154	20.542	0.08	-	0.05
5	2H-benzimidazol-2-one	148	26.895	-	5.21	-
6	methanone	182	33.521	-	0.20	-
	total			<u>0.32</u>	<u>5.41</u>	<u>0.07</u>
	n			<u>4</u>	<u>2</u>	<u>2</u>
<i>Sulfur-containing compounds</i>						
1	disulfide	146	15.175	0.11	-	0.03
	total			<u>0.11</u>	<u>0.00</u>	<u>0.03</u>
	n			<u>1</u>	<u>0</u>	<u>1</u>

(continued)

Table 6 (continued)

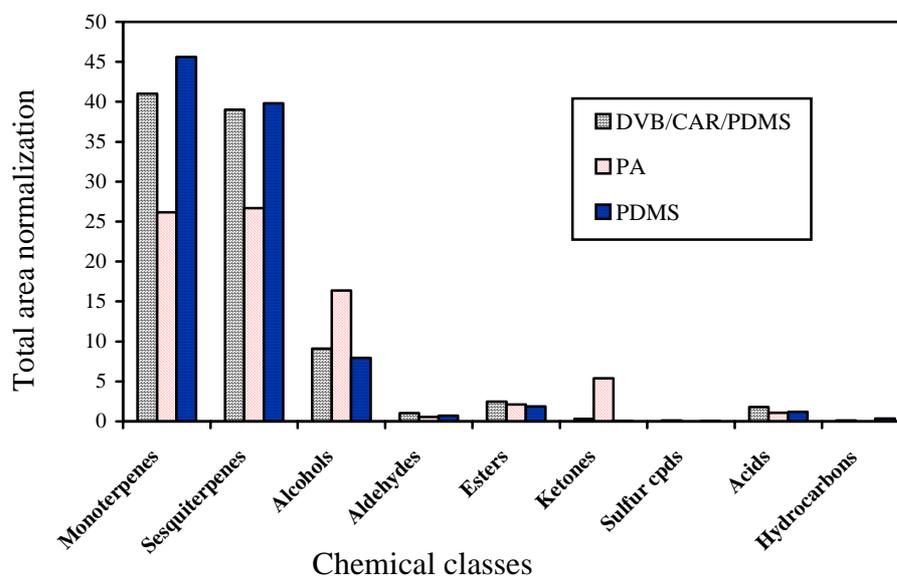
No.	Compounds	MW	$t_R$ (Min)	% Relative peak area		
				DVB/CAR/PDMS	PA	PDMS
<i>Carboxylic acids</i>						
1	ethylic acid	60	2.153	0.06	0.08	0.02
2	acetic acid	196	24.134	0.72	0.17	0.39
3	2,6-octadienoic acid	182	25.442	0.92	0.21	0.42
4	dodecanoic acid	200	32.054	-	-	0.07
5	tetradecanoic acid	228	36.647	-	-	0.13
6	pentadecanoic acid	242	38.734	-	0.13	-
7	2-propenoic acid	290	39.431	0.08	-	-
8	n-hexadecanoic acid	256	40.706	-	0.50	0.18
	total			<u>1.78</u>	<u>1.08</u>	<u>1.20</u>
	n			<u>4</u>	<u>5</u>	<u>6</u>
<i>Hydrocarbons</i>						
1	styrene	104	7.317	0.02	-	-
2	9-ethoxyphenanthrene	222	36.945	-	-	0.16
3	A-phellandrene epoxide	152	22.297	0.08	-	-
4	caryophyllene oxide	220	32.694	-	-	0.18
	total			<u>0.10</u>	<u>0.00</u>	<u>0.34</u>
	n			<u>2</u>	<u>0</u>	<u>2</u>
88	Total identified			94.95	78.36	97.37
	Total identified compounds per fiber			61	44	64

A total of 61 compounds were identified with DVB/CAR/PDMS fiber, amounting to 95% of the total volatile. The sample was dominated by the monoterpene hydrocarbons fraction (41%), with sabinene (33%) and  $\alpha$ -phellandrene (4%) the main compounds. The sesquiterpene hydrocarbons fraction was fewre (39%) with  $\alpha$ -farnesene (15%), isosativene (8%), and trans- $\beta$ -farnesene (7%) as the main components.

It could identify 44 compounds with PA fiber, amounting to 78% of the total volatile. The sample was dominated by monoterpene and sesquiterpene hydrocarbons at approximately the same percentage relative area of 26–27%. Sabinene (22%), and  $\alpha$ -farnesene (12%) were the main components. The alcohols fraction represented 16% of the total volatile with phenol (7%) as the major compound.

Sixty-four compounds could be identified with PDMS fiber, amounting to 97% of the total volatile. The sample was dominated by the monoterpene hydrocarbons fraction (46%) with sabinene (37%) and  $\alpha$ -phellandrene (4%) the main compounds similar to DVB/CAR/PDMS fiber.

The distribution of chemical classes (monoterpene hydrocarbons, sesquiterpene hydrocarbons, alcohols, aldehydes, esters, ketones, sulfur-containing compounds, carboxylic acids, and hydrocarbons) of aroma compounds in mafai jeen fruit obtained through the usage of three fiber coatings are shown in Figure 4. PDMS



**Figure 4** Distribution of chemical classes of aroma compounds in Mafai Jeen fruit by DVB/CAR /PDMS, PA, and PDMS fibers.

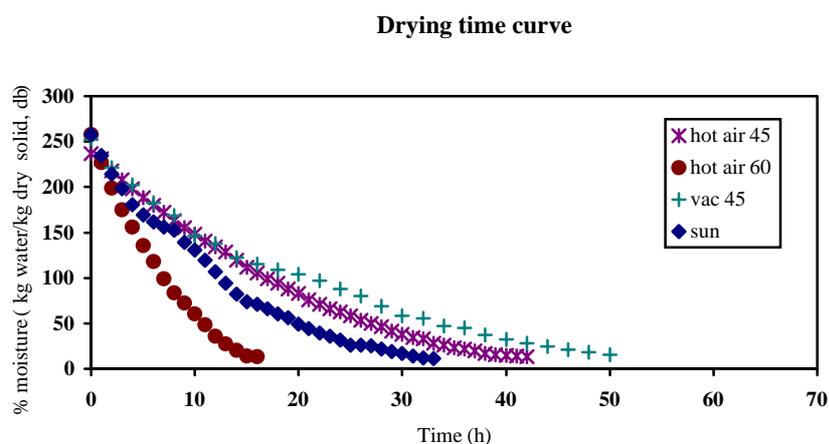
and DVB/CAR/PDMS showed a slightly different pattern among aroma compounds detected (Table 6 and Figure 4). The percentage of alcohols and ketones distributed in PA was higher than in PDMS and DVB/CAR/PDMS.

HS-SPME is an appropriate tool for qualitative and quantitative analysis of aroma compounds that varied according to the fiber coating used. Therefore, it is necessary to carefully select the fiber coating depending on the objective of the study.

## 2. To Study Effect of Drying Methods on the Mafai Jeen Qualities

### 2.1 Effect of Drying Conditions on Qualities of Dried Mafai Jeen

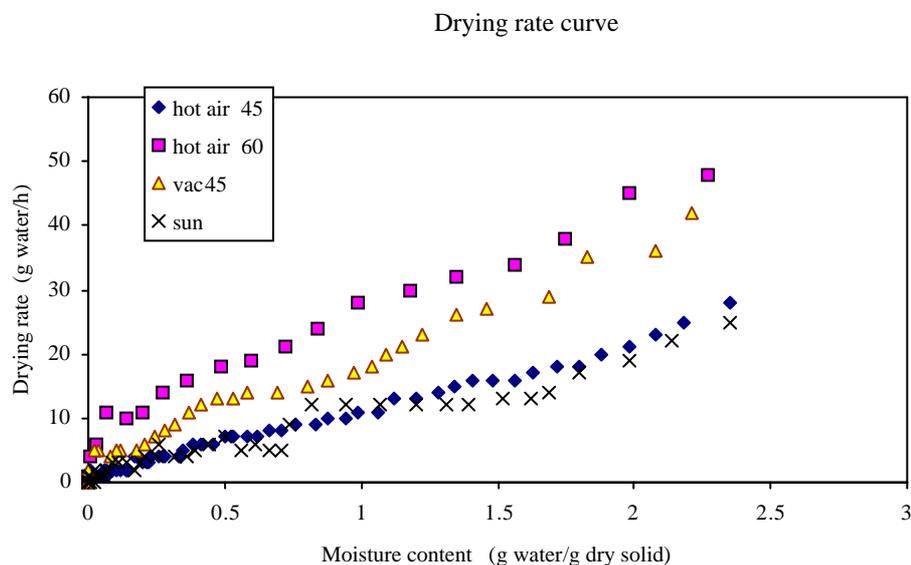
In this study, the effect of drying method on drying rate of Mafai Jeen was evaluated. It was found that visual appearance (attractive color) of vacuum dried Mafai Jeen sample was better than sun dried and hot air dried samples (see appendix Figure C1). When the experimental data of moisture content vs drying time were plotted, concave downward curves were obtained. These are typical of the drying curves obtained during drying. The results were generally in agreement with some literature studies on drying of various food products (Madamba *et al.*, 1996; Senadeera *et al.*, 2003; Yaldiz and Ertekin, 2001). Figure 5 shows the various drying



**Figure 5** Drying time curve at different drying methods (sun drying, vacuum drying at 45 °C, hot air drying at 45 °C, and hot air drying at 60 °C).

methods. It can be seen that the increase in temperature from 45 °C to 60 °C hot air drying, reduced the time needed to reach equilibrium moisture content. This is according to kinetic theory, due to the increased energy of water molecules as temperature was increased. Hence, escaping of molecules became easier and faster from the medium.

Drying time in order to reach moisture content of about 14% wet basis (0.16 kg water/kg dry solid) was 17, 32, 42, and 50 h by hot air drying at 60 °C, sun drying, hot air drying at 45 °C, and vacuum drying at 45 °C, respectively. This moisture content was selected since it is the final moisture content of commercial dried Mafai Jeen product. It has also been speculated (Karel *et al.*, 1994) that moisture contents at or below 15% (wet basis) for most fruit is a rather safe indication that there is no microbial or mould growth and the reaction rate of a number of other deteriorative reactions (sugar crystallization, non-enzymatic browning, flavor deterioration, lipid oxidation, etc) is significantly reduced.



**Figure 6** Drying rate curves for Mafai Jeen at different drying methods ( sun drying, vacuum drying at 45 °C, hot air drying at 45 °C, and hot air drying at 60 °C).

The drying rate was determined from the slopes of the moisture content vs drying time curves, at each measurement point. The variation of the drying rates

against moisture content are shown in [Figure 6](#) for sun dry, vacuum dry at 45 °C, hot air dry at 45 °C, and hot air dry at 60 °C. It was found that the drying rate decreases continuously with decreasing temperature. A constant rate period was observed in sun drying of the Mafai Jeen samples. A comparison of the drying rates for the drying method obtained in experiment were hot air drying at 60 °C higher than vacuum drying at 45 °C, hot air drying at 45 °C and sun drying respectively.

### 2.1.1 Changes in Product Color

Non-enzymatic reaction or the Maillard reaction, is often the limiting factor of dehydrated foods, particularly those with intermediate moisture content (Marty-Audouin *et al.*, 1999). The Maillard reaction occurs when foods are heat-treated. Parameters affected in the Maillard reaction are primarily temperature and the duration of heat treatment. The retention of color can be a quality indicator to evaluate the extent of deterioration due to thermal processing (Avila and Silva, 1999). Change of color and total change value are shown in [Table 7](#).

**Table 7** Changes of color  $L^*$   $a^*$   $b^*$  values and total change ( $\Delta E$ ) for Mafai Jeen at different drying methods (sun drying, vacuum drying at 45 °C, hot air drying at 45 °C, and hot air drying at 60 °C).

Samples	Color change			
	$\Delta L^*$	$\Delta a^*$	$\Delta b^*$	$\Delta E$
Sun drying	-10.43	4.06	-6.10	12.75
Vacuum drying, 45 °C	-4.96	3.83	-2.10	6.61
Hot air drying, 45 °C	-12.07	2.10	-6.16	13.72
Hot air drying, 60 °C	-18.02	0.25	-11.71	21.49

Compared to the initial values  $L^*$   $a^*$   $b^*$  ( 38.95, 5.6, 17.94) of Mafai Jeen fresh fruit before drying, the  $\Delta L^*$  values of hot air drying at 60 °C, hot air drying at 45 °C, sun drying, and vacuum drying at 45 °C decreased considerably to -18.02, -

12.07, -10.43, and -4.96, respectively. The respective net changes in product color,  $\Delta E$ , were observed to be 21.49, 13.72, 12.75, and 6.61 for the same condition as  $\Delta L^*$  values. Vacuum drying at 45 °C had 6.61  $\Delta E$  giving the best color dried Mafai Jeen but took the longest time to be dried. Sun drying had 12.75  $\Delta E$  which was a reasonable good color and also had a reasonable time of drying (32 h).

### 2.1.2 Proximate Analysis

The results of the chemical analysis performed on fresh and sun-dried fruit of Mafai Jeen are summarized in [Table 8](#) The measure pH value of the fruit juice was 3.3, which corresponds with the high concentration of fruit acids and total soluble solid was 17.5 °Bx.

**Table 8** Proximate composition of fresh and dried Mafai Jeen fruits.

Components ( % by wt)	Fresh fruits	Sun-dried fruits
Water	71.97	16.34
Carbohydrate (by difference)	18.64	58.38
Fat	0.33	0.39
Fiber	4.58	8.42
Protein	1.88	7.56
Ash	2.6	8.91

### 2.1.3 Sensory Analysis

Sensory analysis scores of dried Mafai Jeen fruits are shown in [Table 9](#). Mafai Jeen sample from sun drying was rated significantly higher than hot air drying and vacuum drying for aroma, flavor, texture and overall acceptance, except for color from vacuum drying which scored 7.6 or close to ‘Like very much’ and was higher than sun drying. The difference was particularly high with the special aroma of sun-dried fruit.

**Table 9** Mean values for sensory evaluation of dried Mafai Jeen from different drying methods (sun dried, hot air dried at 45 °C, hot air dried at 60 °C, and vacuum dried at 45 °C).

Attributes	Sun-dried	Hot air dried		Vacuum dried
		45 °C	60 °C	
Color	6.8 <sup>b</sup>	6.9 <sup>b</sup>	5.4 <sup>c</sup>	7.6 <sup>a</sup>
Aroma	6.7 <sup>a</sup>	5.9 <sup>b</sup>	6.3 <sup>ab</sup>	5.9 <sup>b</sup>
Flavor	7.7 <sup>a</sup>	7.0 <sup>b</sup>	6.2 <sup>c</sup>	5.5 <sup>d</sup>
Texture	7.2 <sup>a</sup>	6.8 <sup>a</sup>	5.6 <sup>b</sup>	5.6 <sup>b</sup>
Overall acceptance	7.1 <sup>a</sup>	6.4 <sup>b</sup>	5.8 <sup>c</sup>	5.9 <sup>c</sup>

Mean in the same horizontal row with different superscripts are significantly different ( $p < 0.05$ ).

## 2.2 Aroma Components of Mafai Jeen Fruit Treated by Different Drying Condition

The volatile fractions from Mafai Jeen fruit samples were isolated by hydro-distillation to obtain aroma compounds. They were transparent yellowish oils in the appearance, lighter than water and having a floral-fresh-lemon odor. the essential oil yield during extraction from vacuum dried sample compared to the oil obtained from

**Table 10** Yields of oil in fresh fruit and dried Mafai Jeen by sun dried, vacuum dried at 45 °C, hot air dried at 45 °C, and hot air dried at 60 °C.

Sample No.	Fruit/sample	Moisture(%)	Essential oil (ml/100 g)	
			Dried fruit basis	Fresh fruit basis
1.	Fresh	72.05	0.11	0.11
2.	Sun-dried	15.75	0.50	0.16
3.	Vacuum dried at 45 °C	15.50	0.62	0.20
4.	Hot air dried at 45 °C	14.85	0.35	0.11
5.	Hot air dried at 60 °C	15.40	0.35	0.12

fresh fruit was about 82% higher. The yield of oil from Mafai Jeen fruit was 0.11 and 0.62 ml for 100 g fresh and vacuum dried fruits, respectively (Table 10). In order to study the effect of drying method on volatile compounds of Mafai Jeen fruit, the volatile compound in the essential oils of the fresh fruit was compared with these found in different drying method. Using the mass spectral matching against library standard. The results are shown in Table 11.

Fifty-three compounds could be identified in all 5 essential oils of Mafai Jeen fruit, using GC-MS with a HP-5MS column which represent about 97.40-99.07% of the total relative area. The Mafai Jeen fruit essential oil consists a mixture of monoterpene hydrocarbons, sesquiterpene hydrocarbons, alcohols, aldehydes, ketones, carboxylic acids, and terpene oxides. No ester was detected in the examined fruit samples. All of Mafai Jeen oils were nearly similar in their main compositions. They consist mainly of monoterpene hydrocarbons ( fresh fruit; 94.48%, hot air dried at 45 °C; 92.59%, hot air dried at 60 °C; 92.88%, vacuum dried at 45 °C; 94.13%, and sun-dried; 94.05%). The monoterpene hydrocarbons in fresh fruit, hot air dried at 45 °C, hot air dried at 60 °C, vacuum dried at 45 °C, and sun-dried oil were distinctly dominated by sabinene constituting about 66.73, 33.68, 41.13, 64.48 and 63.18% of the oils' composition, respectively. It is accompanied by significant amounts of  $\alpha$ -pinene (11.74, 12.33, 12.02, 13.35, and 9.57%),  $\alpha$ -phellandrene (7.25, 5.77, 6.15, 7.79, and 10.76%), and myrcene (4.27, 4.07, 4.50, 3.65, and 3.20%). Except of 2 samples; hot air dried at 45 °C and hot air dried at 60 °C were contained large amount of  $\beta$ -phellandrene 33.34 and 25.06% respectively. The aroma of fruits can change on heating due to the liberation of aroma substances from glycosidic precursors, oxidation, water addition, and cyclization of individual

**Table 11** Volatile compounds identified in the Mafai Jeen essential oils; fresh fruit, hot air dried at 45 °C, hot air dried at 60 °C, vacuum dried at 45 °C, and sun-dried in order their retention time ( $t_R$ ) using HP-5MS non-polar column.

Compounds	$t_R$ (Min)	% Relative peak area				
		Fresh fruit	Hot air 45 °C	Hot air 60 °C	Vacuum 45 °C	Sun- dried
<i>Monoterpene hydrocarbons</i>						
1. d-limonene	9.029	-	-	-	-	0.03
2. tricyclene	9.144	-	-	-	0.04	-
3. $\alpha$ -thujene	9.433	-	-	0.04	0.05	-
4. $\alpha$ -pinene	9.750	11.74	12.33	12.02	13.35	9.57
5. camphene	10.274	0.54	0.73	0.65	1.09	0.76
6. $\beta$ -pinene	11.520	-	-	-	-	0.10
7. myrcene	12.390	4.27	4.07	4.50	3.65	3.20
8. $\alpha$ -phellandrene	12.939	7.25	5.77	6.15	7.79	10.76
9. $\delta$ -3-carene	13.145	-	-	0.22	-	-
10. 3-carene	13.150	0.22	0.20	-	-	-
11. 4-carene	13.511	1.92	1.21	1.66	1.65	3.58
12. sabinene	14.400	66.73	33.68	41.13	64.48	63.18
13. $\beta$ -phellandrene	14.526	-	33.34	25.06	-	-
14. $\beta$ -fenchene	16.112	0.04	-	0.02	-	0.03
15. $\gamma$ -terpinene	19.373	1.60	1.26	1.24	1.88	2.49
16. $\alpha$ -fenchene	19.676	0.17	-	0.19	0.15	0.18
17. trans-ocimene	27.380	-	-	-	-	0.17
	total	94.48	92.59	92.88	94.13	94.05
<i>Sesquiterpene hydrocarbons</i>						
1. 1H-3a, 7-methanoazulene	29.606	-	-	-	0.04	0.19
2. isosativene	29.851	0.13	0.10	0.07	0.30	0.19
3. bergamotene	30.303	-	-	-	0.03	-

(continued)

Table 11 (continued)

Compounds	$t_R$ (Min)	% Relative peak area				
		Fresh fruit	Hot air 45 °C	Hot air 60 °C	Vacuum 45 °C	Sun- dried
4. tran- $\beta$ -farnesene	30.924	-	-	-	-	0.05
5. naphthalene	31.443	-	0.10	0.05	-	-
6. $\alpha$ -muurolene	31.534	-	0.03	-	-	-
7. curcumene	31.650	0.03	-	-	-	-
8. $\beta$ -biabolene	32.304	-	-	0.04	-	-
9. $\beta$ -farnesene	32.314	-	-	-	0.04	-
10. cis-calamenene	32.679	-	0.04	-	-	-
11. $\delta$ -cadinene	32.679	-	-	0.02	-	-
12. valencene 2	34.208	-	-	-	0.03	-
13. aromadendrene	35.076	-	-	-	0.04	-
14. $\gamma$ -curcumene	36.329	-	-	-	-	0.04
	total	0.16	0.27	0.18	0.48	0.47
<i>Alcohols</i>						
1. 1-pentanol	7.576	-	0.05	-	-	-
2. 1-octanol	7.610	-	-	0.03	-	-
3. linalool	17.584	0.38	-	0.31	0.16	0.24
4. Fenchol	18.084	0.18	0.23	0.21	0.12	0.13
5. Isoborneol	20.590	0.05	-	0.06	-	0.08
6. 3-cyclohexen-1-ol	21.128	0.80	0.85	0.99	0.94	1.04
7. p-menth-2-en-1-ol	21.407	0.07	-	-	-	0.04
8. $\alpha$ -terpineol	21.835	1.18	1.23	1.17	1.00	1.79
9. Santalol	27.077	-	-	-	0.21	-
10. limonyl alcohol	27.076	0.07	-	0.07	-	-
	total	2.73	2.36	2.84	2.43	3.32

(continued)

Table 11 (continued)

Compounds	$t_R$ (Min)	% Relative peak area				
		Fresh fruit	Hot air 45 °C	Hot air 60 °C	Vacuum 45 °C	Sun- dried
<i>Aldehydes</i>						
1. $\alpha$ -campholene aldehyde	17.295	0.06	-	-	-	-
2. Benzaldehyde	24.023	-	-	0.05	-	-
3. Phellandral	25.278	-	0.07	-	0.08	0.19
total		0.06	0.07	0.05	0.08	0.19
<i>Ketones</i>						
1. Pulegone	20.984	0.13	-	-	0.15	-
2. 2-cyclohexen-1-one	21.566	1.02	1.34	1.07	0.90	0.43
3. Carvota acetone	24.244	-	-	-	-	0.05
4. Piperitone	24.518	0.08	-	0.13	-	-
total		1.23	1.34	1.20	1.05	0.48
<i>Carboxylic acids</i>						
1. acetic acid	25.591	0.13	0.11	0.07	0.14	0.17
total		0.13	0.11	0.07	0.14	0.17
<i>Terpene oxides</i>						
1. cis-linalool oxide	16.208	-	0.07	-	0.08	-
2. $\alpha$ -pinene oxide	17.295	0.06	-	-	-	-
3. cis-limonene oxide	19.041	0.10	0.13	0.09	0.10	-
4. phellandrene epoxide	22.378	0.12	-	0.09	0.09	0.12
total		0.28	0.20	0.18	0.27	0.12
Total identified		99.07	96.94	97.40	98.58	98.80

compounds (Belitz and Grosch, 1999) Monoterpene hydrocarbons are common components of traditional foods occurring in essentially all fruits and vegetables. *d*-limonene,  $\beta$ -myrcene, and terpinolene are currently recognized by the U.S. Food and Drug Administration (FDA) as GRAS (“generally regarded as safe”) for their

intended use as flavoring substances (Hall and Oser, 1965].  $\beta$ -myrcene occur naturally in a wide variety of foods including lemon peel oil, orange peel oil, orange juice, and lime juice.

The major alcohols were  $\alpha$ -terpineol (1.00-1.79%), 3-cyclohexen-1-ol (0.80-1.04%) and fenchol (0.13-0.23%). Only one compound of the sesquiterpene hydrocarbon; isosativene was presented in all of samples in small amounts (0.07-0.30%)

In the fresh fruit, it could identify 27 components in this oil, amounting to 99.07% of the relative area. Ten monoterpene hydrocarbons, 2 sesquiterpene hydrocarbons, 7 alcohols, 1 aldehyde, 3 ketones, 1 carboxylic acid, and 3 terpene oxides were found. The monoterpene hydrocarbons fractions (94.48%) dominated the essential oil sample. Sabinene (66.7%),  $\alpha$ -pinene (11.7%),  $\alpha$ -phellandrene (7.2%), and myrcene (4.3%) were the major components. The oxygenated components represented 4.3% of the total oil with  $\alpha$ -terpineol (1.18%) and 2-cyclohexen-1-one (1.02%) as the main components. The sesquiterpene hydrocarbons were smaller with isosativene (0.13%) and curcumene (0.03%).

Three components; curcumene (0.03%),  $\alpha$ -campholene aldehyde (0.06%), and  $\alpha$ -pinene oxide (0.06%) were lost or changed to other compounds after drying.

Thirteen components remaining after different drying method were;  $\alpha$ -pinene, camphene, myrcene,  $\alpha$ -phellandrene, 4-careen, sabinene,  $\gamma$ -terpinene, isosativene, fenchol, 3-cyclohexen-1-ol,  $\alpha$ -terpineol, 2-cyclohexen-1-one, and acetic acid.

Probably, some of the identified compounds were formed or produced during drying and extraction of the samples, specially alcohols, and aldehydes. No relationship was found between the retention of volatile compounds by fresh fruit and different drying process. After vacuum drying, 7 new compounds; tricyclene (0.04%),

1H-3a,7-methanoazulene (0.04%), bergamotene (0.03%),  $\beta$ -farnesene (0.04%), valencene2 (0.03%), aromadendrene (0.04%), and santalol (0.21%) were produced.

After sun drying, 6 new compounds; d-limonene (0.03%),  $\beta$ -pinene (0.1%), trans-ocimene (0.17%), tran- $\beta$ -farnesene (0.05%),  $\gamma$ -curcumene (0.04%), and carvota acetone (0.05%) were produced. After hot air drying at 60 °C, 5 new compounds;  $\delta$ -3-carene(0.22%),  $\beta$ -biabolene (0.04%),  $\delta$ -cadinene (0.02%), 1-octanol (0.03%), and benzaldehyde (0.05%) were produced.

After hot air drying at 45 °C, 3 new compounds;  $\alpha$ -muurolene (0.03%), cis-calamenene (0.04%), and 1-pentanol (0.05%) were produced.

Earlier compositional studies of wild *C. lansium* essential oils in Hainan Island, China were found phellandrene (54.8%), limonene (23.6%), and *p*-menth-1-en-4-ol (7.5%) in the seed,  $\beta$ -santalol, 9-octadecenamide and sinensal in the flowers (Zhao *et al.*, 2004). Therefore, many of the volatile compounds identified using GC-MS are being reported for the first time.

### **3. To Study the Intensity of Astringent Taste of Mafai Jeen**

Initially, the individual TI curves for each of the 9 assessors for each of 6 samples were examined; a total of 54 curves. In [Figure 7](#) a selected set of curves corresponding to tannic acid sol. 1 g/l, 25 °C. are presented for each assessor. The shapes of the curves are basically of 3 types:

a. sharply curve, fast increasing from zero to maximum intensity, I max = 121 mm of astringency in 10s followed by decreasing as a normal curve (example, assessor 3, 4)

b. slow increasing to maximum intensity of astringency, 20s followed by slow decreasing at a rate of 24/s (example, assessor 9)