Effect of extraction and concentration processes on properties of longan syrup

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Abstract Longan (*Dimocarpus longan* Lour.) syrup is a novel liquid sweetener produced from longan, one of the traditional and economic fruits in the Northern of Thailand. In this research, the effect of extraction and concentration processes on properties of longan syrup was investigated. There were two extraction methods (juice extractor and hydraulic press) and three concentration methods (direct heating, steam heating and vacuum evaporation). Results overall showed that the extraction method had no significant ($p \geq 0.05$) effect on longan syrup properties, while concentration resulted in the quality changes of longan syrup. Concentration using direct heating of longan juice caused reduction of sucrose content, and longan syrup dark in color. The headspace volatile compounds of longan syrup were sampled using direct headspace technique and further characterized using gas chromatography-mass spectrometry. The identified volatile compounds could be divided into two groups of aroma characteristics which were (1) floral aroma: 3-methylbutyl acetate, (β)-ocimene and 2-phenylethyl alcohol and (2) caramel aroma: butyraldehyde, furfural and benzaldehyde. 2-Phenylethyl alcohol, contributing to floral odor, was retained using vacuum evaporation as a concentration method. Result revealed that the optimal concentration process for longan syrup production was vacuum evaporation, providing the highest floral volatile and lowest caramel volatile. Sensory tests confirmed

that longan flavor of the syrup produced from the vacuum evaporation process had significantly higher hedonic scores than other processes.

Keywords Longan syrup · Headspace analysis · Extraction · Concentration · Sensory evaluation

Introduction

Longan (*Dimocarpus longan* Lour.) is one of the traditional and economic fruits in the northern part of Thailand, especially in Lamphun and Chiang Mai provinces. Longan is a non-climacteric fruit which is sweet, juicy and aromatic (Li et al. 2009). It can be consumed as fresh or processed fruits, for example, longan in can and dried longan (Lapsongphol and Mahayothee 2007). The major market of fresh longan and its processed products is China. In 2011, Thailand exported longan and longan products about 483,000 t and the total value of longan product was about 406.3 million \$US (Office of Agricultural Economic Thailand 2012). Even fresh longan is aromatic, but its aroma is mild. There are four volatile compounds of fresh longan fruit, i.e., ethanol, ethyl acetate, and trans and cis-(β)-ocimene, while there are more compounds developed during drying process of longan (Lapsongphol et al. 2007). These volatiles are, for example, 3-methyl butanol, 3-methyl butanal and phenyl ethyl alcohol (Lapsongphol et al. 2007; Zhang et al. 2009).

With high total soluble solid of fresh longan about 18–25°brix, it is gradually accepted by consumers over the world due to its sweet juicy mouthfeel and health benefits (Tongdee 1997; Rangkadilok et al. 2007). Most of the carbohydrates in longan are in the form of fructose, glucose and sucrose, which are easily absorbed by the human body. The main sugar compositions of pre-concentrated longan juice are 2.77 % glucose, 3.91 % fructose and 14.21 % sucrose as reported by

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Yunchalad et al. (2008). Therefore, longan juice can potentially be used for syrup production. Recently, there has been production of longan syrup, a new and high commercial product in market. Longan syrup is considered to have functional properties such as the ability to act as a sugar replacer and use for fructo-oligosaccharides production (Surin et al. 2012). Although there are some reports about extract longan juice (Yunchalad et al. 2008), optimization study of extraction and concentration processes have been further required, especially the influence of flavor and aroma. Main processes for longan syrup production are extraction of longan juice and concentration. Juice extraction is the process which the liquid part is separated from the solid part. It can be done using a press or an extractor (Oyeleke and Olaniyan 2007). During this process, juice is exposed to atmosphere which could cause deterioration to juice properties, consequently to longan syrup (Li et al. 2009). The pressing is the most used method for juice extraction, because it is conventional method, i.e., juicer, screw extraction, hydraulic press and burr machine (Beveridge 1997; Barwal and Shreru 2009). The aim of extraction is to obtain the maximum juice yield and high nutrition (Schilling et al. 2007).

For the concentration process, concentration of fruit juices not only provides microbiological stability, but also provides economical packaging, transportation and distribution of the final products (Vijayanand et al. 2010; Fazaeli et al. 2013). Fruit concentration is generally achieved by vacuum evaporation, particularly in case of heat-sensitive food. The process is carried out using low pressure and temperature for evaporation. Therefore, the obtained products have high quality compared to the conventional concentration method using high temperature for evaporation. This is due to the temperature used in conventional evaporation was usually higher than boiling water, resulting in loss of nutrition and low in quality (Keshani et al. 2010). The processes could affect aroma and flavor of longan products, which are important attributes for consumer purchasing and acceptance. Therefore, in this research, we aimed to study the effect of extraction and concentration processes on physical properties, volatile compounds, sugar contents and sensory properties of longan syrup. Two extraction processes of juice extractor and hydraulic press, and three concentration methods of direct heating, steam heating and vacuum evaporation were evaluated.

Materials and methods

Preparation of longan syrup

Fresh longan fruits (cultivar Edor) were purchased from the local market in Chiang Mai Province, Thailand. The longan fruits, 1 kg for each treatment, were washed in tap water two times, and then they were peeled and seeded manually. A 2×3 factorial in completely randomized design (CRD) was used in this

experiment with two main factors i.e. extraction and concentration, which were performed in triplicate. Longan juice was extracted by a juicer (753, Moulinex, Spain) and a hydraulic press (B1, Sakaya, Thailand). The obtained juice was boiled for 15 min and then filtrated through four-layer cheesecloth (20 mesh). Afterwards, the juice was concentrated using three different methods, direct heating, steam heating using steam jacket and vacuum evaporation using rotary vacuum evaporator (BUCHI, Switzerland) at 80 mmbar. All concentration methods were operated at 80 °C to reach final total soluble solid of longan syrup at 60 °brix. The operating temperatures of direct heating and steam heating were controlled using a digital thermometer (Hanna, Thailand). For vacuum evaporation, the temperature was controlled by the automatic system set by the equipment. The operating times of the direct heating, steam heating and vacuum evaporation were about 50 min, 60 min and 70 min, respectively. The completion of concentration was judged according to the final soluble solid of 60 °brix. The percentages of production yield were calculated from longan juice.

Water activity, pH and color measurement

Water activity (a_w) was measured at 25 °C using an AquaLab (Model series 3, Decagon Device Inc., USA). A digital pH meter ((F-22) Horiba, Japan) was used for pH measurement. The color (CIE L^* , a^* and b^*) values of the syrup were determined using Hunter LAB (Colorquest Xe, Hunter Associates Laboratory, USA).

Analysis of sugar contents using high performance liquid chromatography (HPLC)

An HPLC (Agilent series 1200, Waldbronn, Germany) coupled with a refractive index (RI) detector was used to evaluate sugar contents in longan syrup produced by six different processes. The samples were diluted in HPLC grade water (RCI-Labscan, Thailand), afterwards they were filtered through a 0.45 µm membrane disc. The prepared samples, 20 µL, were injected into a Rezex RSO-Oligosaccharides column (Ag^+ form, 200×100 mm, Phenomenex, Torrance, USA). The analysis was operated at 40 and 32 °C for the column oven and the detector, respectively. The flow rate of mobile phase, HPLC grade water, was controlled at 0.25 mL/min. Standard solutions of sucrose, glucose and fructose (Sigma Chemical Co., USA) were used for interpretation and quantification of sugars and their contents in the longan syrup.

Analysis of volatile compounds using gas chromatography-mass spectrometry (GC-MS)

Direct headspace technique was used in this experiment. Before sampling of headspace VOCs the samples were equilibrated at 80 °C for 30 min. Then, 1 mL of headspace were

sampled and directly injected in the injection port. To analyze the volatile compounds, a gas chromatograph (GC, HP7890A, Agilent Technologies, Paolo Alto, USA) coupled to a mass spectrometer (MS, 5975C, Agilent Technologies, Paolo Alto, USA) were used. The volatiles were separated in an HP-5MS column (30 m×0.25 mm, i.d., 0.25 µm film thickness). The operating conditions were started at a temperature of 35 °C for 4 min, then heat up to 200 °C with a heating rate of 7.5 °C/min, finally increase 200 to 250 °C with a heating rate 40 °C/min and kept for 2 min. The detector temperature was 270 °C. Helium was used as a carrier gas at a flow rate of 1.2 mL/min. The mass spectrometer was operated in the scan mode in a range of 34–300 amu, a source temperature of 270 °C, and EI mode at 70 eV. The library of standard mass spectra of the National Institute of Standards and Technology (NIST, Gaithersburg, USA) was used to interpret the chromatograms. The results were reported as relative values of peak area to the one which had the lowest peak area. The calculation was done by comparing the same compound detected in longan syrup produced from different methods. The peak areas of the compound were divided by the peak area of the one has the smallest amount (the lowest peak area).

Sensory evaluation

The consumer preference of longan syrup was evaluated using 9-point hedonic scale, where 1 = dislike extremely, 5 = neither like nor dislike and 9 = like extremely (Resurreccion 1998). The evaluated attributes were color, sweetness and longan flavor which is important parameters for consumer acceptance. The evaluation procedure was carried out according to Stone and Sidel (1995) and Lim (2011). There were 50 consumers involved in this study. The test was done in a sensory evaluation room with Susense program (Silpakorn University, Nakorn-pathom, Thailand) at the Sensory Evaluation and Consumer Testing Unit, Faculty of Agro-Industry, Chiang Mai University, Chiang Mai, Thailand. This room was built according to the ISO 8589-2007-Sensory analysis-General guidance for the design of test rooms. The temperature of the room was controlled at 25 °C. The consumers were sitting in separated booths. For each test, 15 mL of longan syrup were served in a plastic cup and closed with a plastic lid. The random of three digits was applied to randomize the sample for evaluation. The palate cleaning materials were water and cracker, which were served interval of each test. The experiment was done in triplicate. The results were calculated to mean and standard deviation.

Statistical analysis

The data, three replications, were statistically analyzed using SPSS version 16.0 (SPSS Inc., Chicago, USA). The statistical significance of the results was evaluated by one-way analysis

of variance (ANOVA) and Tukey's test with 95 % significance level.

Results and discussion

Yield percentage, water activity, pH and color measurement

Longan syrup was analyzed for their physiochemical properties and compared with the longan syrup extracted by different methods (juicer and hydraulic press), which concentrated by direct heating, steam heating and vacuum evaporation. Table 1 shows that the yield percentages of longan syrup produced from the six methods were not significant difference ($p \geq 0.05$), ranging from 10.2 to 14.2 %. As well the water activity values of the syrup were not significantly different. It has previously shown that longan juice is low acidic with a pH close to 6.0, due to the compositions of organic acids, i.e., gluconic acid, malic acid and citric acid (Li et al. 2004). In our samples after longan juice was concentrated, the pH of the syrup obtained by direct and steam heating became lower, ranging from 5.32 to 5.93, while the ones from vacuum evaporation showed an increase of pH, equal to pH 6.37 and 6.39, in case of hydraulic press and juicer extraction, respectively. In case of direct heating it is possible to consider that higher heating process may lead to lower pH because of sugar caramelization. Moreover, when sucrose is heated above 180 °C the glycosidic bonds of sucrose are hydrolyzed to glucose and fructose and there is a release of water. After that intramolecular rearrangement occurs and hydrogen ions (H^+) are released (Coca et al. 2004). This mechanism may explain why samples of longan syrup obtained by direct heating showed a decreased pH. Although the heating temperature of longan syrup was controlled at 80 °C, heat spots could have occurred, particularly where the matrix was close to the heating source. On the other hand, glucose and fructose contents did not show a statistical increase in case of direct heating process, therefore the reason of a lower pH of these samples cannot be clearly linked to the sole hydrolytic of sucrose. Other mechanisms are probably involved.

Longan syrup color was determined using Hunter LAB, reported L^* (100 lightness; 0 darkness), a^* (+ redness; –greenness) and b^* (+ yellowness; –blueness). The L^* , a^* and b^* values of longan syrup obtained from vacuum evaporation were significantly ($p < 0.05$) higher than those of direct heating and steam heating, which lightness yellow. In contrast, direct heating and steam heating had red-brown color with L^* , a^* and b^* values of 23.2–26.6, 2.7–3.6 and 3.5–5.2, respectively. This is probably due to sugar molecules were developed to unsaturated ring with double bonds in a molecule, and thus light absorption was changed, leading to brown color (Fennema 1996; BeMiller 2007). Longan syrup from direct heating and steam heating processes were red-brown color. In

Table 1 Yield percentages, water activity, pH and color of longan syrup produced from different extraction and concentration methods

Processes		% Yield ^{ns}	Water activity ^{ns}	pH	Color		
Extraction	Concentration				L*	a*	b*
Juicer	Direct heating	10.2±0.64	0.875±0.003	5.93±0.04 ^b	26.6±0.01 ^b	3.7±0.01 ^c	3.1±0.03 ^a
	Steam heating	14.2±0.50	0.891±0.001	5.86±0.01 ^b	24.4±0.01 ^{bc}	5.2±0.02 ^{bc}	3.6±0.05 ^a
	Vacuum evaporation	11.7±2.12	0.887±0.000	6.39±0.02 ^a	30.1±0.01 ^a	7.1±0.02 ^{ab}	0.8±0.08 ^b
Hydraulic press	Direct heating	10.3±0.76	0.881±0.013	5.53±0.00 ^c	23.2±0.01 ^c	3.5±0.05 ^c	2.7±0.05 ^a
	Steam heating	11.9±0.01	0.891±0.000	5.32±0.01 ^c	23.6±0.26 ^c	3.9±0.03 ^c	3.2±0.02 ^a
	Vacuum evaporation	12.2±1.43	0.889±0.002	6.37±0.03 ^a	29.2±0.20 ^a	7.4±0.14 ^{ab}	0.8±0.06 ^b

The data shown were mean ± SD of three replications

The different letters in the same column mean significantly different ($p<0.05$)

ns indicates no significantly different ($p\geq0.05$)

addition, direct heating of longan syrup caused Maillard reaction and caramelization, leading to syrup darker in color (Wang et al. 2006). The browning of product was also found in another type of fruit syrup such as date syrup. The mechanism of non-enzymatic browning is from melanoidin and phenolic compounds in the material (Nasehi et al. 2012).

Analysis of sugar contents using high performance liquid chromatography (HPLC)

There are three types of sugar contained in longan syrup, which are sucrose, glucose and fructose. There were no effects of extraction and concentration processes on glucose and fructose contents ($p\geq0.05$), while concentration processes significantly affected sucrose contents ($p<0.05$). It can be seen in Table 2 that concentration of longan juice using direct heating (177.24–192.55 g/L) led to the reduction of sucrose content because of higher temperature and lower pH,

Table 2 Sugar contents of longan syrup produced from different extraction and concentration methods

Processes		Sugar contents (g/L)		
Extraction	Concentration	Sucrose	Glucose ^{ns}	Fructose ^{ns}
Juicer	Direct heating	177.2±2.21 ^c	101.8±1.47	61.0±3.26
	Steam heating	219.3±3.56 ^a	87.3±18.01	68.0±9.15
	Vacuum evaporation	219.0±1.15 ^a	124.9±14.66	66.7±5.33
Hydraulic press	Direct heating	192.5±2.90 ^{bc}	120.5±21.09	67.0±2.64
	Steam heating	215.5±0.52 ^{ab}	116.1±19.09	76.9±5.61
	Vacuum evaporation	240.5±14.91 ^a	139.2±15.04	86.4±18.06

The data shown were mean ± SD of three replications

The different letters in the same column mean significantly different ($p<0.05$)

ns indicates no significantly different ($p\geq0.05$)

followed by steam heating (215.53–219.29 g/L) and vacuum evaporation had highest sucrose (218.97–240.53 g/L). The contents of glucose and fructose were, however, not increased, indicating that Maillard reaction took place. Moreover, caramelization could occur (Wang et al. 2006). Therefore, syrup concentrated by direct heating and steam heating contained less sucrose content than the one of vacuum evaporation. Sucrose in product was hydrolyzed to be monosaccharides which were glucose and fructose and released carbon dioxide (CO₂) at high temperature and long time (Fennema 1996). The glucose and fructose were rearranged intramolecule to long chain and released hydrogen ion (H⁺) to system, were lower pH (Coca et al. 2004). These reactions lead to decrease of nutrition value because of sucrose loss (Fennema 1996).

Volatile compounds of longan syrup

Direct headspace technique was used for sampling volatile compounds released from longan syrup. Then they were identified by GC-MS (Figs. 1 and 2). Table 3 shows the list of 13 volatiles detected in headspace of longan syrup, and their relative amounts by comparing with the ones which presented the least for that compound. Five compounds belong to ester group. They are ethyl 2-butenate, 3-methylbutyl acetate, ethyl nonanoate, ethyl dodecanoate, and phenyl 2-hydroxybenzoate. There are four aldehyde compounds presented, i.e., butyraldehyde, furfural, benzaldehyde, and (*E*)-2-dodecen-1-al. In addition, there are two alcohols of 1-octen-3-ol and 2-phenylethyl alcohol, and two isoprenes of α -pinene and β -ocimene. Generally, these volatile compounds lead to typical aroma of longan syrup that is floral and caramel aroma. Among these volatiles, β -ocimene contributes to typical aroma of longan (Lapsongphol and Mahayothee 2007), and 2-phenylethyl alcohol contributes to floral aroma (Zhang et al. 2009). These two characteristic volatiles of longan syrup were affected by concentration process. For β -ocimene, steam

Fig. 1 Chromatograms of longan syrup produced by different methods; **a** juicer: direct heating; **b** juicer: steam heating; **c** juicer: vacuum evaporation; **d** hydraulic: direct heating; **e** hydraulic: steam heating; **f** hydraulic: vacuum evaporation. *TIC* total ion current

heating maintained the highest content of this compound followed by vacuum evaporation and direct heating. Floral aroma, 2-phenylethyl alcohol, was retained only in longan syrup concentrated by vacuum evaporation. This compound is relatively high volatility; therefore, it is easily evaporated under concentration conditions without vacuum. Butyraldehyde, furfural and benzaldehyde, contributing to caramel

aroma, were found in higher amount in syrup obtained from direct heating, followed by steam heating and vacuum evaporation. These compounds were not detected in fresh longan (Lapsongphol and Mahayothee 2007). They were formed from Maillard reaction using reducing sugars as precursors (Scarpellino and Soukup 1993). The characteristic aroma of longan syrup, it can generally be divided into two groups, that

Fig. 2 Mass spectra fingerprints of volatile compounds identified in longan syrup

Table 3 Relative amounts of the headspace volatiles of longan syrup produced from different extraction and concentration methods

Volatiles	LRI	Juicer			Hydraulic press			Flavor description ^[Ref.]
		Direct heating	Steam heating	Vacuum evaporation	Direct heating	Steam heating	Vacuum evaporation	
Butyraldehyde	813	2.18	1.94	1.00	2.64	2.00	1.21	Chocolate ^a
Furfural	846	1.95	1.20	1.00	2.20	2.11	1.09	Bread, almond ^b
Ethyl 2-butenate	862	1.27	1.49	3.86	1.10	1.00	1.37	Fermented ^a
3-methylbutyl acetate	904	1.52	1.63	4.78	1.00	1.28	5.39	Fruity ^a , banana ^b
Benzaldehyde	993	1.61	1.47	1.00	1.80	1.46	1.38	Almond, burnt sugar ^b
1-octen-3-ol	1011	1.22	1.00	5.08	2.54	1.00	3.84	Earthy ^a , mushroom ^b
(α)-pinene	1042	1.49	1.00	1.53	1.67	1.55	1.66	Herbal ^a
(β)-ocimene	1055	1.00	3.43	2.65	1.47	3.42	2.38	Floral ^a
(<i>E</i>)-2-dodecen-1-al	1091	nd	2.68	nd	1.01	2.19	nd	Citrus ^a
2-phenylethyl alcohol	1097	nd	nd	1.00	nd	nd	1.42	Honey, spice, rose, lilac ^b
Ethyl nonanoate	1270	1.25	2.29	4.53	1.00	2.76	3.21	Waxy ^a
Ethyl dodecanoate	1464	1.29	1.00	2.14	1.29	1.48	1.60	Mango-like ^c
Phenyl 2-hydroxybenzoate	1665	1.82	1.61	2.84	1.88	1.00	2.63	Balsamic ^a

LRI calculated linear retention indices, nd not detected

^a The goods scents company (2011); ^b Acree and Arn (2004); ^c El-Sayed (2012)

is, floral and caramel aroma due to the volatile compounds mentioned before.

Sensory evaluation

Sensory evaluation showed that the color, sweetness and flavor of longan syrup produced from different concentration and extraction processes showed significantly different ($p < 0.05$). Table 4 shows that there were no significant ($p \geq 0.05$) effects of juice extraction on sensory properties of

longan syrup. Consumers preferred longan syrup produced by vacuum evaporation with higher score than the ones of direct heating and steam heating. They obviously liked its color and flavor. This is due to consumers preferred syrup light in color and syrup rich in floral flavor more than caramel flavor. It is consistent with the flavors identified by GC.

Conclusion

Extraction using juicer and hydraulic press did not affect physical and sensory properties, volatile compounds and sugar contents of longan syrup. In contrast, concentration by direct heating, steam heating and vacuum evaporation affected those important features of longan syrup. Vacuum evaporation provided longan syrup with better flavor and these were the only syrups which contained considerable amounts of 2-phenylethyl alcohol. We suggested that this compound may strongly contribute to enhance the consumer preference, although a direct link between this compound and consumer preferred was not verified in this research. Sensory evaluation showed that consumers preferred longan syrup concentrated by vacuum evaporation not only because of their flavor, but also because of their color and sweetness. In addition, it can be concluded that consumers liked syrup with high floral aroma, but low caramel aroma. Future work is required to apply the longan syrup in other food products and find the process to reduce dark color of syrup. Mechanistic pathways involved in

Table 4 Sensory scores obtained from 9-point hedonic scale of longan syrup

Processes		Color	Sweetness	Longan flavor
Extraction	Concentration			
Juicer	Direct heating	5.1±1.98 ^{bcd}	5.7±1.77 ^{abc}	4.9±1.71 ^b
	Steam heating	5.4±1.64 ^{bc}	4.9±1.77 ^c	5.9±1.72 ^a
	Vacuum evaporation	6.5±1.64 ^a	6.0±1.88 ^{ab}	6.0±1.67 ^a
Hydraulic press	Direct heating	4.4±1.60 ^d	5.3±1.72 ^{bc}	5.2±1.62 ^{ab}
	Steam heating	5.0±1.88 ^{cd}	5.8±1.77 ^{ab}	5.8±1.86 ^a
	Vacuum evaporation	6.8±1.55 ^a	6.2±1.61 ^a	5.8±1.84 ^a

The data shown were mean ± SD of three replications ($n=50$)

The different letters in the same column mean significantly different ($p < 0.05$)

the synthesis of volatile compounds of longan fruits should also investigate.

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