

Changes in Volatile Components and Sugar Content of Arenga Sap (*Arenga pinnata* Sap) during the Ohmic Heating Process

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Abstract This research examines the changes in volatile components and sugar content in arenga sap (AS) through the ohmic heating process to produce arenga sap liquid sugar (ASLS). Ohmic heating was performed under various treatments of frequency, voltage, power, and heating time. Meanwhile, AS is frozen sap that has been evaluated with an initial pH of 6 and a total soluble solid (TSS) of 12%, indicating normal fragrance and taste characteristics. Gas chromatography-mass spectrometry (GCMS) was employed to analyze volatile components, while high-performance liquid chromatography (HPLC) was employed to quantify sugar content. According to the research findings, the volatile compound content in AS is primarily composed of ester compounds with diverse derivative components, including N-Heterocyclic (pyrazines), Aldehydes, Ketones, Phenols, and even siloxanes. Despite the fact that the types of volatile components vary in each treatment with ohmic heating, most of them are more complex than AS prior to the heating procedure. Although the sugar was subjected to high temperatures and electric currents during the ohmic heating process, the sugar composition of AS and ASLS did not undergo a significant change, suggesting that sugar degradation did not occur in a drastic manner.

Keywords Arenga sap, GCMS, Liquid Sugar, Ohmic Heating, Volatile Component

1. Introduction

Arenga sap (AS) is the sap from the aren palm tree (*Arenga pinnata*) with a total sugar content ranging from 13.9-14.9% [1] and a productivity of 12% when processed into sugar. Compared to sugarcane, which has a productivity of only 7%, this productivity is regarded as high [2,3]. In addition, AS is also rich in organic acids such as ascorbic acid, lactic acid, acetic acid, citric acid, pyroglutamic acid, and fumaric acid, as well as several volatile compounds, particularly from heterocyclic, non-heterocyclic, aldehyde, and ketone groups [4]. These volatile components will affect the distinctive aroma of the sap (both coconut sap, nipa sap, and AS have different distinctive aromas depending on the composition of their volatile components).

Traditionally, AS has been consumed directly or processed into sugars such as moulded sugar, coconut sugar, granulated sugar or liquid palm sugar. This process will require several hours at temperatures over 100°C until a thick consistency is achieved, which is followed by a change in colour to a darker shade and a unique aroma as a result of two critical reactions: the Maillard reaction and caramelization [5]. The process will also harm and

diminish the amount of volatile components in the sap, reducing its functional characteristics.

In the Maillard reaction, amino acids and reducing carbohydrates (glucose and fructose) are involved in the production of a brown colour effect and an aroma as a result of the changes in characteristic volatile components. This reaction is commonly known as the Strecker degradation [6,7]. However, due to AS's low protein concentration of approximately 0.2% [6], this study will focus on the modifications caused by the caramelization reaction. In the meantime, the caramelization reaction is the decomposition of simple sugars and polymerization that takes place during the heating process [8,9]. The caramelization reaction, like the Maillard reaction, provides sweet properties in ASLS; so, while they occur concurrently, they contribute differentially to the ASLS flavour and scent. In addition to changes in sugar content, this reaction will alter functional qualities due to the breakdown of sugar components in the sap. These changes will influence the characteristics of the ASLS as a sweetener with a low glycemic index (less than 55) [8].

Ohmic heating is an alternate technique that involves the passage of an electric current through a conductive material, causing it to heat up due to the resistance to the flow of electricity, thereby providing a uniform and rapid heating effect. This method offers advantages over conventional heating by reducing thermal gradients and preserving the quality of the treated material. This technique can transform AS into ASLS through the evaporation process. Ohmic heating also offers several advantages, including reduced processing times and enhanced energy efficiency, when compared to thermal and non-destructive methods such as pulsed electric field (PEF) [9], high-pressure processing (HPP) [10], cold plasma [11], high voltage electric field (HVEF) [12] and microwave vacuum evaporation [13]. With a shorter processing time and the use of heat during the measurement process compared to traditional procedures, the probability of preserving volatile components in AS increases, and various more volatile components created can be further examined. The functional properties of the ASLS from AS can be preserved through ohmic heating, as the changes resulting from the degradation of sugar components are not overly substantial.

This research evaluated the modifications in the composition of sugar and the volatile components of ASLS that result from the application of ohmic heating at a variety of frequencies, voltages, and power levels. Recent study on the volatile components of AS, whether pure or processed into sugar, has not been widely published, resulting in very few comparative studies in this field. The investigation of changes in volatile components reveals a variety of differences in the volatile components of AS from different locations. These variations are due to extrinsic factors such as the environment, altitude, and the tapping processes and techniques used. Identifying the volatile component with the most substantial impact will be challenging, and the analytical method employed will also influence the

detectable volatile components [14,15].

2. Material and Method

The main raw material used was AS from Lombok, West Nusa Tenggara, Indonesia. The AS was utilized with a natural preservative during the tapping process of the fragmen from *purut* tree (*Dysoxylum parasiticum*), which was previously dried at a temperature of 50 ± 2 °C for 60 minutes at concentration 0.92% w/v. The concentration is 0.92% w/v. The sap tapping process is conducted in the afternoon, from 06:00 to 15:00 local time (± 10 hours of tapping), provided that there is no rain during the tapping process and that the water content in the sap is not contaminated before the tapping process occurs.

The AS was filtered using a fabric filter. The filtered AS was used to determine pH, total dissolved solids, and color analysis. The sap used contains a pH range of 6-7, a total soluble solids percentage of 12-15%, and a colour value of 60-65 (Lightness Value, LV). LV was used to measure the quality of AS instead of other hue angles because it is often associated with turbidity. The more turbid the AS, the more it indicated that the fermentation process in AS had already begun. Analysis was also conducted on sensory attributes, especially color and aroma, during the ohmic heating process. This analysis was carried out directly to observe changes in AS throughout the process.

The AS is subsequently stored in hermetically sealed glass containers and frozen at -18°C. As shown in Table 1, the ASLS utilized is the result of the heating of AS using an ohmic heater, as indicated by the treatment. The parameters for frequency, power, voltage, and heating time were selected based on preliminary study. This condition based on the best physicochemical properties (data not shown) of ASLS, so the parameters used can be seen in Table 1.

Table 1. The production of aren sap liquid sugar (ASLS) using ohmic heaters

No	Freq (Hz)	Power (watt)	Voltage (V/cm)	Heating Time (Minutes)
1	150	120	18.8	60
2	150	80	15.2	60
3	250	120	18.4	50
4	250	80	14.8	75

2.1. Gas Chromatography-Mass Spectrometry Analysis

AS and ASLS with an ohmic heating process were tested for volatile components using Gas Chromatography-Mass Spectrometry (GCMS) (Shimadzu GCMS-QP2020NX). Injector temperature 250°C, helium gas was used at a flow rate of 14.6 mL/min at a pressure of 78 kPa. Column temperature 100°C with a flow rate of 1.06 mL/min for 25 minutes. Other GCMS conditions are as follows: scanning

mass range (m/z) 35–500 a.m.u at a rate of 11666 scans/second.

2.2. High Performance Liquid Chromatography Analysis

Analysis of volatile components in AS and ASLS with ohmic heating was conducted using High Performance Liquid Chromatography (HPLC) (Shimadzu RID-20A) with a low pressure gradient pump method, pressure 0 kgf/cm²– 250 kgf/cm². The mobile phase of water at a pressure of 0.45 GPa, methanol at a pressure of 1.25 GPa; acetonitrile at a pressure of 1.20 GPa. Sample injection volume 1 mL/min, oven temperature 60–105°C. The detector uses the RID-20A model with the analytical mode at a temperature of 35°C. The column used is YMC pack NH2 no. 5 with a length of 250 mm, an inner diameter of 4.6 mm, and a particle size of 3 μ m. Peak integration uses the chromatopac algorithm with a width of 5 seconds at a slope of 200 μ V/minute. Identification uses a 5% window with the absolute method, and peak selection uses the nearest peak. The quantitative method uses the external standard method.

3. Result

3.1. Characteristics of Sugar

The analysis demonstrated that fresh aren sap (AS) primarily consisted of other components, including water, minerals, and organic acids (89.77%), with sucrose comprising only 10.01%, and minor amounts of glucose (0.125%) and fructose (0.095%). Ohmic heating treatments significantly elevated the sucrose concentration in the aren sap liquid sugar (ASLS). Treatment at 150 Hz, 120 W, 46 V, for 60 minutes resulted in a sucrose content of 50.818%, while a similar frequency at 80 W and 38 V yielded 50.943%. At 250 Hz, sucrose levels increased further, reaching 51.5% with 120 W, 47 V, over 50 minutes, whereas treatment at 80 W, 37 V, for 75 minutes produced a sucrose content of 49.5%. Overall, ohmic heating notably enhanced the sucrose proportion in ASLS compared to the untreated fresh sap, indicating the potential of this method to improve sugar concentration in aren sap derivatives.

3.2. Volatile Compound

GC-MS analysis identified several volatile components in fresh AS. Pentanoic acid, 4-oxo, ethyl ester was the most abundant compound with a relative percentage area of 9.51%, followed by butanedioic acid, diethyl ester (0.48%). Minor compounds included 5-hydroxymethylfurfural and 2-furancarboxylic acid, ethyl ester, each at 0.16%, furfural at 0.11%, and triethyl citrate at 0.07%. The detected volatile compounds belonged primarily to the groups of carboxylic acids, esters, and heterocyclic compounds.

Whereas GC-MS analysis of ASLS following ohmic heating treatments revealed significant variations in

volatile component profiles. Butanedioic acid, diethyl ester was the predominant volatile across all treatments, ranging from 33.17% to 45.45%. Cyclopropane, 1,2,3-tricarboxylic acid, triethyl ester was also detected at notable levels, particularly reaching 18.70% in Treatment 1 and 10.40% in Treatment 3. Other major volatiles included benzoic acid, ethyl ester (6.28–18.81%) and hexanoic acid, 2-methyl-2-propenyl-, methyl ester (3.00–10.50%). Minor compounds such as benzaldehyde, 2,4-dimethyl- (7.52% in Treatment 2) and phenol, 2-[1,1-dimethylethyl]-4-ethyl- (1.92% in Treatment 2) were identified sporadically. Several esters and ketones contributed to fruity, sweet, and creamy aroma profiles, while N-heterocyclic and siloxane compounds were present in trace amounts.

4. Discussion

4.1. Characteristics of Sugar

In both AS and ASLS, sucrose is the predominant form of sugar, as shown in Figure 1. This is an attribute that is shared by all varieties of sap, and it is responsible for the dominant sweet taste that is found in sap. Furthermore, the caramelization process imparts aroma and colour features to sucrose throughout the heating phase. The sucrose content of AS sap before processing was 10.013%, glucose 0.125%, and fructose 0.095%, whereas other components such as water, protein, organic acids, minerals, and non-sugar components were not discovered in the study.

The sucrose content in ASLS that has undergone heating using an ohmic heater is much higher compared to the sucrose content in AS. The overall sugar content has been changed, with glucose varying from 0.462% to 0.75%, fructose from 0.873% to 1.2%, sucrose from 49.5% to 50.943%, and other components from 46.75% to 48.55%.

This increase is mostly explained by the evaporation process during heating, which reduces the water content and results in a thicker sap consistency. This increase in viscosity indirectly concentrates the sucrose content per unit volume, resulting in a higher measurable concentration.

Moreover, the heating process has an impact on the sensory qualities of the ASLS that is produced, including its aroma and colour. As a result of the caramelization reaction that sucrose undergoes, the colour of the product has a tendency to become darker when the concentration of sucrose is increased. Furthermore, heating can also cause the degradation of complex carbohydrates or polysaccharides, which then break down into sucrose, thereby contributing to the increase in sucrose content in the final product. In the heating process using an ohmic heater, the frequency and duration of heating play an important role in influencing the rate of the hydrolysis reaction of sucrose into simple sugars, particularly glucose and fructose. The research results illustrated in Figure 1 show that the sucrose content of the ASLS is not substantially affected by variations in frequency and voltage in various heating duration combinations.

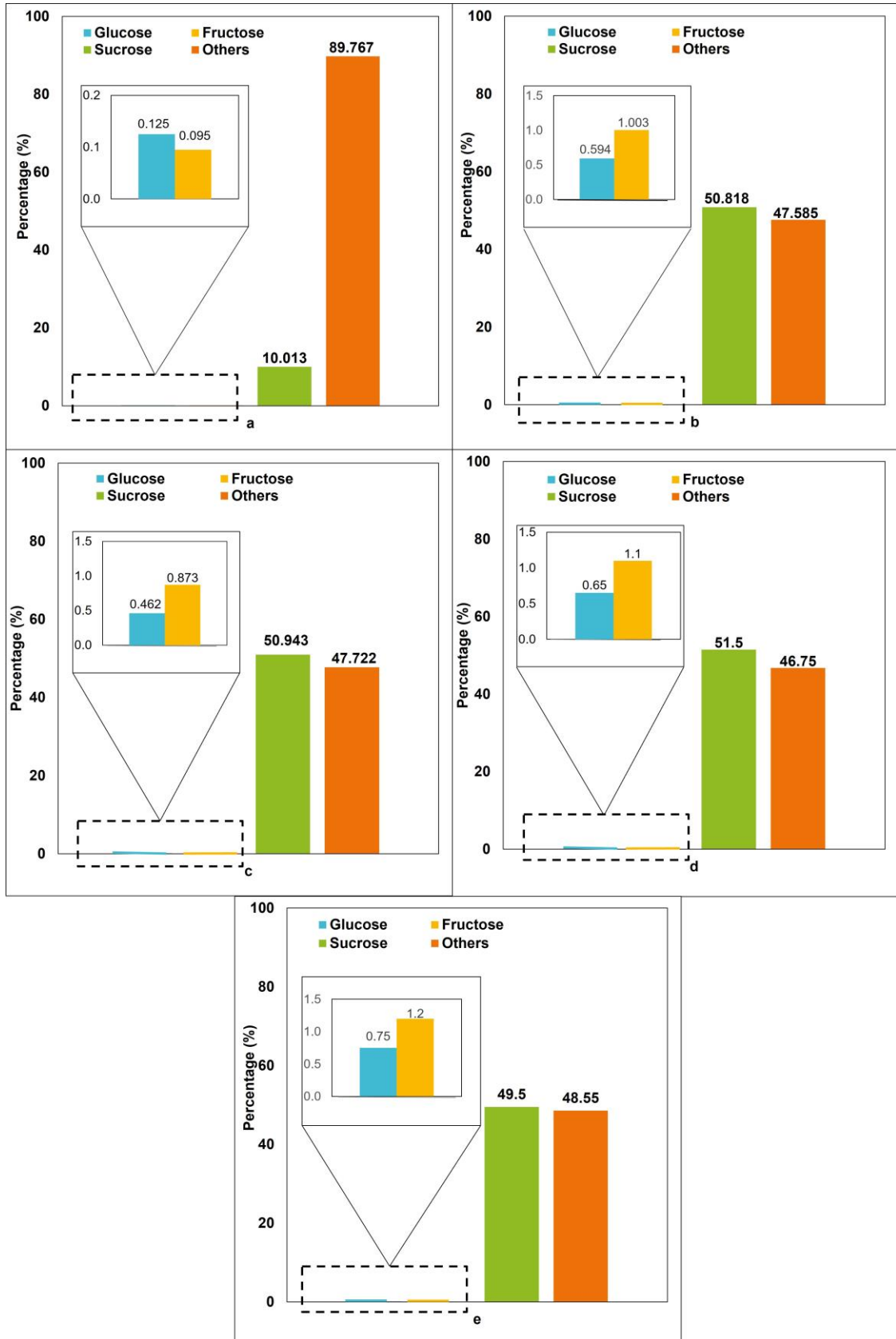


Figure 1. Analyze the characteristics of the components of AS and ASLS (a): AS (Raw); (b) ASLS with ohmic heating treatment at frequency 150 Hz; 120 watt; 18.8 V/cm; heating time 60 minutes; (c) ASLS with ohmic heating treatment at frequency 150 Hz; 80 watt; 15.3 V/cm; heating time 60 minutes; (d) ASLS with ohmic heating treatment at frequency 250 Hz; 120 watt; 18.4 V/cm; heating time 50 minutes; (e) ASLS with ohmic heating treatment at frequency 250 Hz; 80 watt; 14.8 V/cm; heating time 75 minutes)

However, in the treatment with a frequency of 250 Hz, a power of 80 watts, and a voltage of 37 volts for 75 minutes, the sucrose content produced tends to be lower compared to other treatments. This indicates the beginning of the sucrose hydrolysis process, where the compound starts to degrade into glucose and fructose. This indication is reinforced by the significant increase in glucose and fructose levels in that treatment compared to other treatments. Thus, it can be concluded that longer heating durations at high frequencies begin to promote the hydrolysis reaction of sucrose, although not yet optimally, resulting in only a partial change in the total sucrose content.

At the same frequency with different voltages, the amounts of glucose and fructose tend to be higher, indicating a stronger heating effect. At higher frequencies, although the power and voltage used are lower, the longer cooking time allows for reactions that optimize sugar conversion. Based on the ratio of sucrose, glucose, and fructose in ASLS, and assuming the presence of other unmeasured components, such as the formation of proteins or more complex carbohydrates, it can be said that ASLS produced from ohmic heating tends to have a low glycemic index (GI) due to the higher fructose content, which does not raise blood sugar levels as quickly as glucose. The low glycemic index (GI) indicates that the produced sugar is sweet but is absorbed slowly in the body's metabolism, thus helping to control blood sugar spikes [19,20].

4.2. Volatile Compound

The process of making ASLS with ohmic heating, with relatively faster and more uniform cooking times, causes several volatile components in AS to degrade into other volatile components and even new volatile components to emerge. In raw AS, it shows the presence of volatile components such as N-Heterocyclic, O-Heterocyclic,

carboxylic acids, and esters, with the compound Pentanoic acid,4-oxo,ethyl ester dominating up to 9.51% (Table 2). These compound components primarily contributed to the intensity of the characteristic fresh aroma, taste, and are one of the quality components of AS. The other components, including furfural, 2-furancarboxylic acid, ethyl ester, butanedionic acid, diethyl ester, 5-hydroxymethylfurfural, and triethyl citrate, have an approximate consistency in percentage, with each area percentage being less than one percent.

Meanwhile, in the ASLS, under various treatments, the compound Butanedionic acid, diethyl ester dominates (33.18 - 45.45%) (Table 3). This compound is not commonly found in sugar products but may occur due to secondary reactions during the heating process. This secondary reaction involves carbohydrate degradation and esterification, but from Table 2, it can be seen that in AS, the ester composition is very low, so it is possible that carbohydrate degradation is the largest source. Carbohydrates in the AS sourced from simple sugars sucrose, glucose, and fructose undergo caramelization reactions during the heating process and degrade into organic acid components such as butanedionic acid, diethyl ester (diethyl succinate), especially due to the presence of Hydroxymethylfurfural compounds that degrade into succinic acid and through esterification reactions into diethyl succinate [21]. This is consistent with the results shown in Figure 1, where at a frequency of 150 Hz, power of 80 watts, voltage of 15.2 V/cm, and at a frequency of 250 Hz, power of 120 watts, voltage of 18.8 V/cm, the sucrose composition is higher compared to other treatments, resulting in higher levels of butanedionic acid and diethyl ester compounds as well. Various compounds show an equivalent ratio, with a maximum percentage of 25%. This includes constituents from the ester compound Hexadecanoic acid, ethyl ester, as well as certain volatile components identified at the amount under 5%.

Table 2. Volatile components, relative percentage area (%) and description of volatile components using GCMS on AS

Volatile components	Relative percentage area (%)	Group of compounds	Aroma description
Furfural	0.11	N-heterocyclic	Sweet, caramel [15]
Pentanoic acid,4-oxo,ethyl ester	9.51	Carboxylic acid	The distinctive aroma of fruits with a sweet and fresh nuance [16]
2-furancarboxylic acid, ethyl ester	0.16	O-heterocyclic	The distinctive aroma of fruits with a sweet and fresh nuance. This aroma can also have a slight woody or balsamic characteristic [17]
Butanedionic acid, diethyl ester	0.48	Carboxylic acid	The distinctive aroma of fruits with a sweet and fresh nuance [16]
5-hydroxymethylfurfural	0.16	N-heterocyclic	Caramer and <i>nutty</i> [15]
Triethyl citrate	0.07	Ester	The aroma of fruits and like oranges [18]

Table 3. Volatile components, relative percentage area (%) and description of volatile components using GCMS on ASLS

Volatile components	Relative percentage area (%)				Aroma description
	Treatment				
	1	2	3	4	
N-Heterocyclic (pyrazines)					
2-Propanone, 1-(1H-pyrazol-1-yl)-	0.00	0.00	0.00	1.44	Nutty [22]
Aldehydes					
Benzaldehyde, 2,4-dimethyl-	0.00	7.52	0.00	0.00	Sweet, caramel [15]
Ketones					
Pentanoic acid,4-oxo,ethyl ester	6.21	0.00	0.00	0.00	fruity & ester; creamy & buttery [23]
Heptanoic acid, 6-oxo-, ethyl ester	0.00	0.00	3.018	0.00	
Ester volatil					
Diethyl methylsuccinate	0.61	0.00	0.00	0.00	The distinctive aroma of fruits with a sweet and fresh nuance [16]
Diethyl fumarate	3.83	0.00	5.43	3.59	
Benzoic acid, ethyl ester	6.28	18.81	9.054	18.18	The distinctive aroma of fruits with a sweet and fresh nuance. This aroma can also have a slight woody or balsamic characteristic [17]
Butanedionic acid, diethyl ester	33.18	45.45	36.12	33.97	
Hexanoic acid, 2-methyl-2-propyl-, methyl ester	8.51	0.00	5.03	13.40	
Ethyl m-methylbenzoate	3.83	4.08	0.00	5.74	
Benzeneacetic acid, ethyl ester	2.84	6.58	2.92	5.02	
Ethyl 4-methylbenzoate	2.91	0.00	3.52	0.00	
Ethyl o-tolylacetate	4.67	0.00	0.00	0.00	
Benzeneacetic acid, .alpha.-methyl-, ethyl ester	1.07	0.00	0.00	0.00	
Benzenebutanoic acid, 2,5-dimethyl-, methyl ester	1.69	0.00	0.00	0.00	
Hexadecanoic acid, ethyl ester	3.75	14.73	21.43	15.79	
Cyclopropane-1,2,3-tricarboxylic acid, triethyl ester	18.70	0.00	13.48	0.00	
Fenol Volatil					
Phenol, 2-(1,1-dimethylethyl)-4-ethyl	1.92	0.00	0.00	0.00	Sweet & spice ^h
Siloksan Volatil					
Cyclononasiloxane, octadecamethyl-	0.00	2.82	0.00	0.00	odorless

1. ASLS with ohmic heating treatment at frequency 150 Hz; 120 watt; 18.8 V/cm; heating time 60 minutes

2. ASLS with ohmic heating treatment at frequency 150 Hz; 80 watt; 15.2 V/cm; heating time 60 minutes

3. ASLS with ohmic heating treatment at frequency 250 Hz; 120 watt; 18.4 V/cm; heating time 50 minutes

4. ASLS with ohmic heating treatment at frequency 250 Hz; 80 watt; 14.8 V/cm; heating time 75 minutes

Based on Table 2, it can be seen that the largest component of the volatile compounds from AS is the carboxylic acid Pentanoic acid, 4-oxo,ethyl ester, which imparts a characteristic fresh and sweet aroma. Meanwhile, the aroma of furancarboxylic acid, ethyl ester detected, in addition to the aroma from the sugar palm trunk, is also suspected to be the aroma from the use of natural preservatives during the tapping process. Meanwhile, Furfural (0.11%) and 5-Hydroxymethylfurfural (HMF) (0.16%) belong to the N-Heterocyclic group, indicating the degradation of sugar in AS, which usually occurs due to several factors such as natural fermentation during the tapping process, exposure to environmental temperatures,

sap acidity, or reactions during the storage process. The content of ester compounds (Butanedionic acid, diethyl ester, and Triethyl citrate) in AS contributes to the distinctive fresh aroma of AS.

The volatile components in ASLS (Table 3) show more varied results due to the caramelization reaction that occurs during the heating process, especially aldehyde and ester compounds. In food products with high sugar content or potentially acidic due to the presence of ascorbic acid, the thermal processing will form furfural and hydroxymethylfurfural compounds, which are hazardous chemical components, especially if the processing is done conventionally where the heat received is not constant and

uncontrolled [22]. However, in the processing of AS using ohmic heaters, these two components were not found, indicating that the heat generated during the processing is more uniform and controlled. At frequencies of 150 Hz and 250 Hz with a higher voltage of 120 watts, more diverse results were obtained, namely 15 volatile components at 150 Hz frequency with 120 watts voltage and 9 volatile components at 250 Hz frequency with 120 watts voltage, with most of them being ester group volatile components.

The volatile component Cyclopropane-1,2,3-tricarboxylic acid, triethyl ester that appears under treatment at 120 watt voltage, both at 150 Hz and 250 Hz frequencies, represents a phenomenon of better heating efficiency. At this voltage, the flow of electrical energy is sufficient to heat the AS in a short time, resulting in the formation of more complex compounds due to the esterification reaction of organic acids with ethanol that may naturally be present in AS [22,24,25]. Whereas at lower voltages, it is not effective enough, so it does not appear during the reading of volatile components. In ASLS, Cyclopropane-1,2,3-tricarboxylic acid, triethyl ester, like other esters, will impart a more complex and sweet aroma to the ASLS. The formed ester can modify the sweetness of the ASLS by adding a new, more complex flavor dimension.

The volatile component of the N-Heterocyclic group (pyrazines), namely 2-Propanone, 1-(1H-pyrazol-1-yl)-, only appears under treatment with high frequency, specifically 250 Hz at 80 watts, whereas at the same frequency with higher voltage it does not appear due to insufficient heating duration. 2-Propanone, 1-(1H-pyrazol-1-yl)- is a compound that appears due to dehydration reactions caused by excessive heating; this reaction will remove water molecules and lead to the formation of new compounds, including carbonyl products. This compound in ASLS will impart aromas such as spice, burnt, and roasty.

Volatile compounds in very small amounts such as Benzeneacetic acid, .alpha.-methyl-, ethyl ester, Benzenebutanoic acid, 2,5-dimethyl-, methyl ester, and phenol, 2-(1,1-dimethylethyl)-4-ethyl are only present in the treatment with a frequency of 150 Hz; 120 watts; 46 volts; heating for 60 minutes, indicating that although using a frequency below 250 Hz can provide more even heat and sufficient time to produce a variety of volatile compounds compared to other treatments. If the time used is slower, there is a possibility that there will not be enough time to form or evaporate the detected compounds, or at too high a frequency, the heat distribution during the heating process is more concentrated in certain areas, thus not supporting the chemical reactions that occur.

5. Conclusions

Based on the overall research results, it shows that there are no noticeable changes in the sugar composition in the treatment of heating using ohmic, whereas compared to AS,

the increase in sucrose occurs mainly due to the heating process that removes water from the material, thereby increasing the concentration of sucrose formed. This indicates that the ASLS formed still has a low glycemic index (GI). Meanwhile, in the testing of volatile components, there is a change from AS to ASLS during the ohmic heating process. The changes that occur are due to the caramelization process and the formation of lipids because of lipid oxidation, or it is necessary to deeply investigate the possibility of strecker degradation due to the amino acid content. The changes in volatile compounds result in a characteristic sweet, roasty, nutty aroma while not losing the natural aroma of ASLS, thus it can be concluded that the ohmic heating process in the production of ASLS does not eliminate the characteristic compounds that preserve the aroma of AS.

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