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Dynamic Changes in Volatile Organic Compounds During the Spoilage of Palm Wine Stored at Ambient Temperature

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A R T I C L E I N F O

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A B S T R A C T

This study aimed to investigate the volatile organic compounds (VOCs) variations during the spoilage of palm wine stored at ambient temperature and identify potential shelf-life markers. Palm wine collected from local tappers and resellers were stored at ambient temperature (28-30°C) for 96 h. At an interval of 24 h, VOCs variations were investigated using Solid phase Microextraction-Gas Chromatography-Mass Spectroscopy (SPME-GC/MS) method. Changes in sensory quality and potential flavour contributors were also explored. The sensory rejection time was found at 24 h and 48 h of storage for palm wines collected from the tappers and resellers, respectively. The first attribute to be spoiled was taste followed by odour and appearance. A total of 23 VOCs distributed in six chemical families were identified. Alteration of palm wine sample is characterised by an increase in concentration of alcohol (isoamyl alcohol, isobutanol and 1-octanol), aceti acid and acetoin, and a decrease in ester concentration (ethyl acetate and ethyl hexanoate). In the view to fight against the short shelf-life and develop new preservation methods, these compounds can be used as markers of spoiled palm wine.

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Introduction

In Côte d'Ivoire, as in several African countries, the sap of tropical Palmae family plants such as oil palm (*Elaeis guineensis*), date palm (*Phoenix dactylifera*) and raffia palm (*Raphia hookeri*) are used to produce a traditional fermented beverages called palm wine or bangui (Karamoko et al., 2012). The palm tree is first uprooted and then left on the ground for one to two weeks. The branches are then pruned and a rectangular hole is drilled at the upper end on the "heart" of the palm tree where the terminal bud is located. The sap is collected in a canary or pot using a bamboo pipe placed in this hole. Cut and trimmed branches are arranged to protect the hole from dust and dirt. Before the palm wine is collected, the palm's "heart" is heated to eliminate the microbial flora and allow the sap to flow easily. The beverage is collected twice a day over a period of four to eight weeks.

The process of tapping of palm wine is a semi-continuous fermentation mainly conducted by yeast and lactic acid bacteria. *Saccharomyces cerevisiae* was found as the dominant yeast species while *Leuconostoc mesenteroides* and *Fructobacillus durionis* were the dominant bacteria (Amoikon et al., 2019; Kouamé et al., 2020). This beverage is very popular and highly appreciated by all consumers. (Karamoko et al., 2012). This traditional wine plays an important role in traditional ceremonies (marriage, birth, baptism, etc.), religious celebrations and is sometimes used as a solvent to extract the active ingredient from plants that can fight malaria (Chandrasekhar et al., 2012). It is also an important source of vitamins, sugars, amino acids and other nutritional compounds that complement the diet of consumers (Erukainure et al., 2019). Furthermore, the relatively higher abundances of gevotroline (an antipsychotic and dopamine D2 receptor antagonist) and mitoxantrone (an anticancer drug) in palm wine support the therapeutic potential of this beverage (Djeni et al., 2020).

However, like most of African traditional alcoholic beverages, palm wine has short shelf-life (Amoa-Awua et al., 2007; Karamoko et al., 2012; Titilayo and Owoola, 2019). In general, Changes in turbidity and viscosity, the formation of sediment and film, as well as bitterness and off-flavours (mousiness, ester taint, phenolic, vinegary, buttery, geranium tones), are all signs of wine deterioration caused by microbial growth (du Toit & Pretorius, 2000). The production of biogenic amines and ethyl carbamate precursors can also be the result of the activity of spoilage micro-organisms (Shiling et al., 2015).

Several authors reported that the diversity of volatile compounds existing in varying concentrations is at the origin of the characteristic flavour that defines the smell and taste of a specific alcoholic beverage (Andrés-Iglesias et al., 2015; Alvim et al., 2017). Such compounds include among others acids, alcohols, esters, aldehydes, furanic compounds and volatile phenols. In this regard, various studies on the impact of variations in volatile compounds on organoleptic characteristics have been carried out for several beverages, including wine (Moyano et al., 2019; Pérez-Jiménez & Pozo-Bayon, 2019), African opaque beer (Ncube et al., 2020), and beer (Shale et al., 2013; Gonzalez Viejo et al., 2019). For palm wine, no investigation has dealt in detail with its fast spoilage kinetic, particularly the volatile compound variations.

The present study sought to investigate the volatile organic compound (VOC) variations during the spoilage of palm wine stored at ambient temperature and identify potential deterioration markers.

Materials and Methods

Samples Collection and Storage Conditions

Palm wine samples were collected from two tappers and two resellers located at Bingerville and Yopougon, respectively (district of Abidjan, Southern Côte d'Ivoire). The criteria used to select these tappers and resellers were their willingness to take part in the study, and the availability and accessibility of production sites. In addition, the individuals selected have at least 5 years' experience in palm wine production and selling. From tappers, samples of fermented sap were taken directly in the collection pot at weekly intervals, namely 1st, 7th, 14th and 21st day of tapping in the early hours of the day (7:00 am). From resellers, the palm wine samples were taken directly from their stock of the trading day. Pre-sterilised 3x500 mL Plexiglas containers were used to collect the samples, which were immediately placed in an isothermal box and transferred to the laboratory within 2 hours. Once at the laboratory, each sample was subdivided into volumes of 200 mL in sterile bottles and stored at ambient temperature (28-30°C) for 96 h. At an interval of 24 h, one bottle was taken out for analysis. All experiments were carried out independently in triplicate.

Physicochemical Analysis

Palm wine samples were characterized for their pH, total titratable acidity (TTA) and total soluble solids (TSS). A digital pH-meter (P107 Consort) was used to determine pH value while TTA was determined by titrating 5 mL of palm wine sample against 0.1 M NaOH using phenolphthalein as the indicator. The TTA was expressed as the percentage of lactic acid (%). TSS content, expressed as °Brix values, was determined using a hand refractometer (IP65: PAL-1, ATAGO). The refractometer has been calibrated with distilled water. For the pH meter, calibration was carried out systematically prior to pH measurements using two buffer solutions at pH=7 and pH=4. All measurements were performed in three replicates.

Sensory Evaluation

The study was reviewed and approved by the Institutional Review Board for research ethics and informed consent was obtained from each subject prior to their participation in the study.

The sensory test was conducted as previously described by Attchelouwa et al. (2017). Briefly, the test consisted of establishing any difference between fresh and stored samples, by a panel of 10 semi-formed judges, who are conventional consumers of palm wine. Two-week training sessions introduced the judges to the rating scale and the sensory attributes to be assessed. At each testing session, the judges were served a plate containing randomized three-digit coded cups with samples. The criteria for their inclusion in the sensory panel were fidelity (repeatability and reproducibility), accuracy and sensitivity. The sensory evaluation was conducted in the sensory evaluation laboratory under normal lighting and at room temperature. The judges scored the samples for odour, appearance, and taste using a scoring scale with three categories corresponding to 1 = fresh (sample similar to fresh palm wine or without any off-odour), 2 = marginal (sample having slight difference or slight off-odour but still being acceptable), and 3 = spoiled (sample largely different from fresh palm wine or with strong off-odour). A fresh palm wine sample was used as the reference. The category of each palm wine sample was defined when at least 50% of the judges evaluated the sample in one of the three categories. The experiment was conducted in triplicate.

Determination of Volatile Organic Compounds

Volatile organic compounds (VOCs) were analysed by Solid phase Microextraction-Gas Chromatography-Mass Spectroscopy (SPME-GC/MS), following the method previously described by Assi-Clair et al. (2019) with slight modifications. One gram of sample, 1 g of NaCl, and 3 µL of 3-heptanol (concentration of 2.454 mg/mL) used as an internal standard (IS) for semi-quantification of VOCs were placed in a 20 mL glass vial. The vial was tightly capped with a PTFE-silicon septum on a heating platform. VOCs were extracted using a 50/30 µm divinylbenzene/carboxene/polydimethylsiloxane (DVB/CAR/PDMS) SPME fiber provided by Supelco. The SPME fiber was conditioned in the chromatograph injector at 250°C for 30 min prior to extraction. The extraction process was carried out at 50°C for 45 min with constant stirring (500 rpm).

Extracted VOCs were analysed using an Agilent 7890 B gas chromatography-mass spectrometer (GC-MS) equipped with a DB-WAX Agilent 122-7062E column, 60 m length x 0.25 mm internal diameter x 0.25 µm film thickness (Palo Alto, CA, USA). The GC oven temperature was initially set at 40°C for 5 min, then raised to 140°C at a rate of 2°C/min and to 250°C at a rate of 10°C/min. The total run time was set at 66 min and injection mode was splitless at 250°C for 2 min. Helium was used as the carrier gas with the flow rate fixed at 1 mL/min. The selective mass detector was a quadrupole (Hewlett Packard, Model 5973), working in electron ionization (EI) mode at 70 eV with a scan range m/z 40-300. The temperatures of the MS transfer line and the ionization source were 150°C and 230°C, respectively. The analysis of each sample was carried out in duplicate.

The identification of VOCs was done by (i) comparing the mass spectra with commercial database Wiley275 or using the NIST mass spectral search program (NIST/EPA/NIH database version 2.0), considering a minimum similarity value of 70%; (ii) comparing the linear retention index calculated (RI) with those found in the literature. The RIs were calculated in the same chromatographic conditions using series of n-alkanes C8-C20 (Supelco, Bellefonte, USA). Peak areas were used for relative quantification using the MSD Chemstation software (version E.02.02.1431, Agilent Technologies).

Statistical Analysis

One-way analysis of variance (ANOVA) and Duncan test were used to compare the physicochemical characteristics and volatile compounds of fresh and deteriorating palm wine samples using the R software 3.6.1 with ''agricolae'' package. Significance was set at $P < 0.05$. Principal Component Analysis (PCA) were performed with VOCs which showed significant fluctuations to compare palm wine samples obtained from different storage time. PCA allowed the measured variables to be grouped into new variables called 'components' or 'factors.' This grouping is based on the correlation of the variables.

Results and Discussion

Changes in physico-chemical characteristics

The values of TSS of the fresh palm wines ranged from 1.4 °Brix to 5.3 °Brix (Figure 1). Except for the sample P21D, TSS values decreased significantly during the first 24 hours of storage. Thus, the values decreased from 5.3 °Brix to 1.9 °Brix and from 2.25 °Brix to 1.6 °Brix, respectively for the samples P1D and P7D. After 24 h of storage, TSS values remained constant until 72 h and they decreased slightly at 96 h of storage. The decrease of TSS is a result of soluble sugars conversion into ethanol and organic acids (Titilayo & Owoola, 2019). The decrease of TSS content is thus attributable to the high activity of microorganisms, as reported by Ojo-Omoniyi et al. (2020). Amoa-Awua et al (2007) also reported a steady decrease in total sugars from 11.02% to 3.32% in palm wine during 24 hours of storage, while yeast, aerobic mesophilic bacteria, lactic acid and acetic acid bacteria loads did not vary significantly.

As shown in Figure 1, pH of the fresh palm wines analysed in this study was acid. The values were ranged from 3.06 (P21D) to 4.54 (P1D). During the storage at ambient temperature, the pH values of wine samples decreased gradually to 2.40-3.48. In contrast to pH, TTA of wine samples increased throughout the storage. The acidity of the wine P21D increased from 2.08% to 3.25% while that of the wine RS2 increased from 0.76% to 1.05% (Figure 1). This could be due to the production of organic acid by microorganisms during palm wine storage. Karamoko et al. (2012) found lactic, acetic and ascorbic acids as the major organic acids and citric, malic, fumaric, oxalic and tartaric acids as the minors.

Changes in the sensory quality

The results of the sensory evaluation of palm wine samples stored for 96 h at ambient temperature are shown in Table 1. For samples collected from tappers (P1D, P7D, P14D and P21D), 50% (mean percentage of six independent trials, each assessed by 10 judges) of the judges noticed that the appearance of the wines was spoiled only after 96 h of storage. On contrary, they scored this attribute as fresh for palm wines collected from resellers throughout the study. The odour also was judged as spoiled according to the wine sample origins. Thus, for samples from resellers, the odour spoilage time was determined at 96 h of storage while for those from palm wine tappers, it was found at 72 h (P7D, P14D and P21D) or at 48 h (P1D). As with odour, more than half of the judges reported that the taste of samples from tappers was spoiled after 24-48 h of storage and after 48-72 h for samples from resellers. The sensory rejection time was found at 24 h and 48 h for samples from tappers and local resellers, respectively. This result agrees with previous reports which showed that the shelf life of African beverages was only 48 hours after production (Lyumugabe et al., 2012; Attchelouwa et al., 2017). According to Lyumugabe et al. (2012), this spoilage is mainly the result of the metabolic activities of mesophilic lactic acid bacteria.

In this study, the taste was the first attribute judged as spoiled, followed by odour and appearance. Attchelouwa et al. (2017) tasted the sensory quality of the spoiling traditional sorghum beer tchapalo. They also found the taste as the first spoiled attribute followed by odour and appearance. This result is in accordance with the fact that taste is the determining attribute in a choice of a particular food for each person (Choi, 2013).

Changes in volatile organic compounds from fresh to spoiled palm wines

A total of twenty three volatile compounds distributed in six chemical families were identified in palm wine samples. These families are terpenes (3 compounds), esters (8 compounds), ketones (5 compounds), alcohols (3 compounds), acids (2 compounds) and aldehydes (2 compounds). Most of compounds detected in the current study have also been reported in deteriorating sorghum beer (Attchelouwa et al., 2020), wine (Ubeda et al., 2016; Moyano et al., 2019; Pérez-Jiménez and Pozo-Bayon, 2019), African opaque beer (Ncube et al., 2020) and indigenous banana beer (Shale et al., 2013).

Table 2 shows VOCs identified in palm wine collected from a local reseller and how they varied over the 48 h storage period. o-cymene was the major compound among the chemical family terpenes. Its concentration in the fresh sample were 14.420 µg/L. The concentrations of terpenes remained statistically constant up to 48 h of storage. Among esters family, the concentration of ethyl acetate which was the major compound decreased up to 48 h of storage. The values passed from 912.247 µg/L to 736.171 µg/L (Table 2). There were also significant variations in the concentrations of isoamyl alcohol, methyl vinyl ketone, ethyl decanoate, ethyl octanoate, ethyl hexanoate, acetoin and acetic acid.

Table 1. Sensory scores (in % of judges) of palm wine during the storage at ambient temperature

Sample	Storage duration	Appearance			Odour			Taste		
		Fresh	Marginal	Spoiled	Fresh	Marginal	Spoiled	Fresh	Marginal	Spoiled
P1D	24 h	65	25	10	40	25	35	05	15	80
	48 h	55	20	25	20	20	60	00	00	100
	72 h	50	20	30	20	10	70	00	00	100
	96 h	30	15	55	10	10	80	00	00	100
P7D	24 h	60	40	00	65	20	15	20	45	35
	48 h	60	40	00	40	30	30	10	30	60
	72 h	55	45	00	15	30	55	00	00	100
	96 h	30	10	60	15	30	55	00	00	100
P14D	24 h	50	25	25	45	40	15	20	40	40
	48 h	45	25	30	30	40	30	05	35	60
	72 h	40	25	35	30	15	55	00	15	85
	96 h	15	30	55	25	15	60	00	10	90
P21D	24 h	75	20	05	40	45	15	35	35	30
	48 h	30	55	15	20	45	35	05	40	55
	72 h	30	55	15	05	40	55	05	35	60
	96 h	15	25	60	05	40	55	00	30	70
RS1	24 h	65	15	20	45	50	05	25	65	10
	48 h	65	15	20	40	40	20	25	60	15
	72 h	50	25	25	15	50	35	05	35	60
	96 h	50	15	35	15	15	70	00	35	65
RS2	24 h	60	30	10	50	40	10	00	60	40
	48 h	65	20	15	35	45	20	05	25	70
	72 h	55	25	20	15	40	45	00	15	85
	96 h	60	15	25	10	25	65	00	00	100

P1D, P7D, P14D, P21D: palm wine samples collected from the tappers at the day 1, 7, 14 and 21 respectively. RS1, RS2: palm wine samples collected from the local resellers 1 and 2, respectively

Table 2. Concentration (µg/L) of volatile organic compounds identified in palm wine sample collected from a local reseller (RS1) and stored at ambient temperature

Chemical group	Compounds	Storage duration (h)		
		0	24	48
Terpenes	o-Cymene	14.420 ± 0.923^{a}	17.117 ± 0.777^{a}	13.372 ± 0.930^{a}
	beta- Myrcene	0.735 ± 0.476^{a}	0.985 ± 0.770^{a}	0.672 ± 0.375^{a}
	D-Limonene	5.544 ± 0.806^{a}	7.161 ± 0.281^{a}	5.127 ± 0.948^{a}
Alcohols	Isobutanol	9.904 ± 0.723^{a}	12.461 ± 1.474^{a}	10.980 ± 1.744^{a}
	Isoamyl alcohol	22.690 ± 1.900^{a}	42.364 ± 5.912^{b}	72.457 ± 4.173^{c}
	1-Octanol	0.026 ± 0.007^{a}	0.631 ± 0.051^{b}	0.750 ± 0.033^{b}
Esters	Ethyl Acetate	912.247 ± 34.427^{b}	881.654 ± 59.482^{b}	736.171 ± 16.080^{a}
	Methyl butanoate	0.335 ± 0.019^{a}	0.358 ± 0.036^{a}	0.315 ± 0.003^{a}
	Ethyl butanoate	2.027 ± 0.660^{a}	2.804 ± 0.436^{a}	3.609 ± 0.964^{a}
	Orthoformic acid, triisobutyl ester	2.821 ± 0.267^{ab}	3.180 ± 0.418^{ab}	3.222 ± 0.921^{ab}
	Ethyl hexanoate	28.196 ± 2.421^{b}	28.336 ± 2.221^{b}	16.222 ± 1.300^{a}
	Ethyl octanoate	10.191 ± 0.723^{a}	25.060 ± 2.904^{b}	21.057 ± 1.373^{b}
	Ethyl decanoate	4.923 ± 0.534^{a}	21.834 ± 1.238^{c}	11.661 ± 0.197^{b}
	Linalyl formate	2.263 ± 0.968^{a}	2.490 ± 0.302^{a}	2.685 ± 0.912^{a}
Ketones	2-Pentanone	3.112 ± 0.908^{a}	2.510 ± 0.971^{a}	3.139 ± 0.934^{a}
	Methyl vinyl ketone	17.237 ± 1.905^{b}	16.756 ± 1.054^{b}	11.861 ± 1.516^{a}
	3-Heptanone	0.480 ± 0.032^{a}	0.476 ± 0.040^{a}	0.485 ± 0.076^{a}
	Acetophenone	3.147 ± 0.047^{a}	3.140 ± 0.821^{a}	3.191 ± 0.991^{a}
	Acetoin	939.380 ± 9.784^{c}	679.161 ± 1.695^{b}	298.908 ± 1.824^{a}
Acids	Acetic acid	380.046 ± 5.054^{a}	440.034 ± 2.061^{b}	384.489 ± 5.032^{a}
	Benzhydrazide	4.459 ± 0.114^{a}	4.284 ± 0.212^{a}	4.383 ± 0.131^{a}
Aldehydes	Hexanal	0.642 ± 0.018^{a}	0.936 ± 0.084^{a}	0.763 ± 0.059
	Benzaldehyde	3.516 ± 0.904^{a}	3.675 ± 0.857^{a}	3.614 ± 0.071^{a}

Means values (n = 3) followed by the same letter in the same line are not significantly different ($p > 0.05$); ND: not detected.

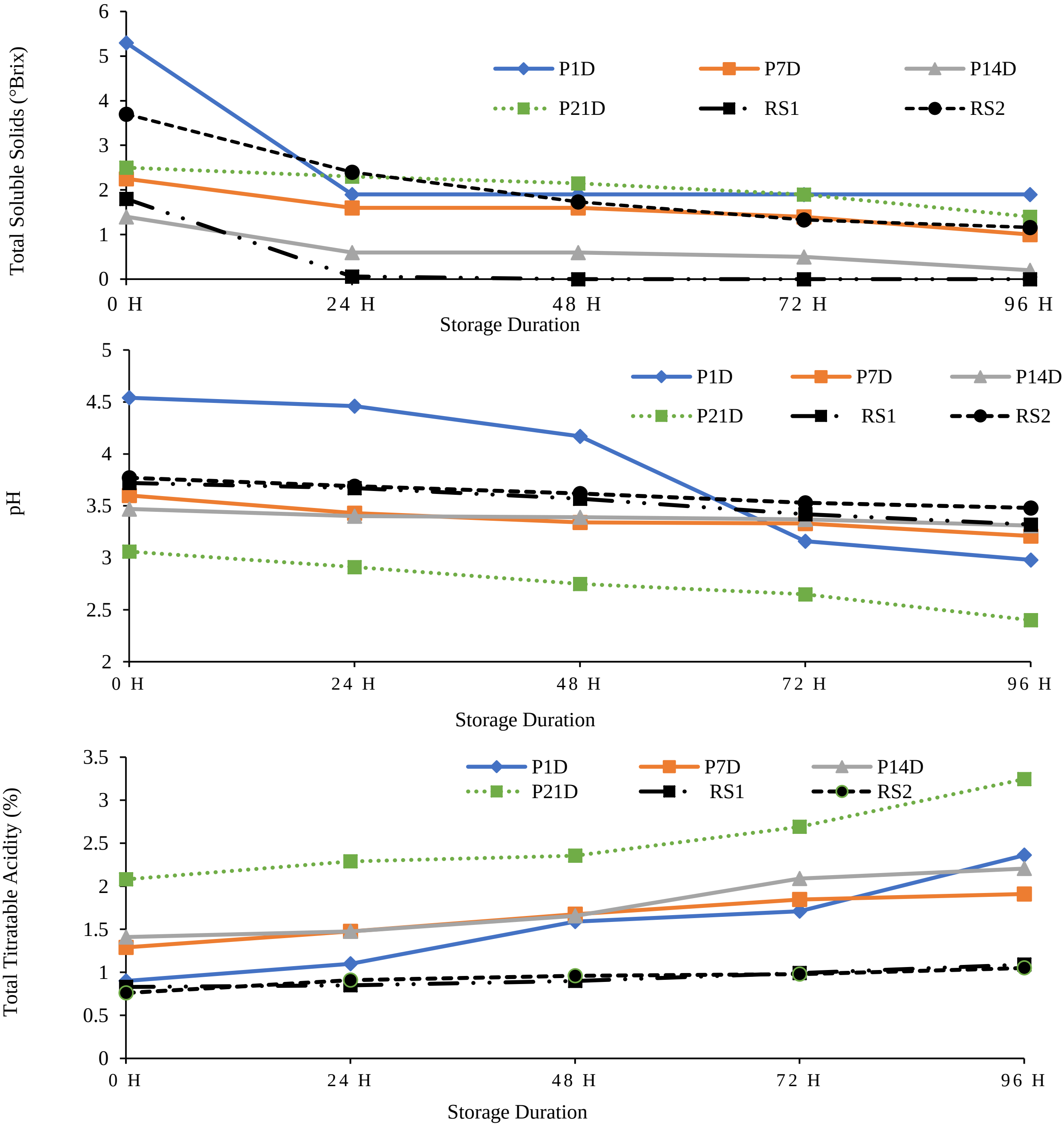

Figure 1. Evolution of the characteristics of palm wine samples during the storage at ambient temperature
P1D, P7D, P14D, P21D: palm wine samples collected from the tappers at the day 1, 7, 14 and 21 respectively. RS1, RS2: palm wine samples collected from the local resellers 1 and 2, respectively

For samples collected at the 1st day of tapping (P1D), ethyl acetate concentration decreased from 3539.776 to 3277.864 µg/L (Table 3). Isobutanol and isoamyl alcohol concentrations significantly increased at 24 h of storage. In samples collected at the 7th day of tapping (P7D), isoamyl alcohol, ethyl acetate, ethyl hexanoate, methyl vinyl ketone and acetic acid concentrations observed significant variations.

Generally, alteration of palm wine sample is characterised by an increase in alcohol concentration (isoamyl alcohol, isobutanol and 1-octanol) and a decrease in ester concentration. Changes in esters concentrations such as ethyl acetate have been reported in wine (Ubeda et al., 2016) and alcoholic bilberry beverages (Lui et al., 2020). The decrease may be due to hydrolysis producing acids while the increase may be a result of the yeasts response to nitrogen availability (Barbosa et al., 2009). The decrease of ethyl hexanoate could result in the loss of the sweet, waxy, green and banana odour. According to Olaniran et al. (2017), an increase in isoamyl alcohol concentration influences drinkability because the beverage flavour becomes heavier. This observation could explain the rejection of palm wine by panelists. In addition, an increase in acetic acid and acetoin concentrations was observed during deterioration. Acetoin and acetic acid come respectively from the oxidation of lactic acid and ethanol by strains of *Acetobacter* and *Gluconobacter* under low-oxygen conditions (Du Toit and Pretorius, 2000). The change in the organoleptic characteristics of the wine, notably the reduction of the fruity character, the increase in acidity and the sherry-like aroma, is associated with an increase in the concentration of acetic acid.

Table 3. Concentration (µg/L) of volatile organic compounds identified in palm wine sample collected from local tappers at the first day of tapping (P1D) and stored at ambient temperature

Chemical group	Compounds	Storage duration (h)		
		0	24	48
Terpenes	o-Cymene	0.464±0.064[a]	0.392±0.016[a]	0.432±0.072[a]
	beta- Myrcene	0.008±0.000[a]	0.008±0.000[a]	0.008±0.000[a]
	D-Limonene	0.336±0.040[a]	0.344±0.080[a]	0.264±0.080[a]
Alcohols	Isobutanol	27.968±0.432[a]	50.816±1.680[b]	48.160±9.016[b]
	Isoamyl alcohol	52.536±12.464[a]	276.192±17.616[b]	266.552±16.696[b]
	1-Octanol	0.048±0.008[a]	0.072±0.008[a]	0.056±0.008[a]
Esters	Ethyl Acetate	3539.776±45.312[c]	3277.864±84.088[a]	3358.472±83.016[b]
	Methyl butanoate	0.352±0.048[a]	0.336±0.008[a]	0.312±0.008[a]
	Ethyl butanoate	2.744±0.136[a]	4.840±0.784[c]	3.608±0.120[b]
	Orthoformic acid, triisobutyl ester	0.960±0.152[a]	1.016±0.160[a]	0.920±0.000[a]
	Ethyl hexanoate	59.160±6.768[b]	103.152±7.616[c]	39.920±3.048[a]
	Linalyl formate	0.432±0.056[a]	0.552±0.064[a]	0.672±0.288[a]
Ketones	2-Pentanone	2.872±0.128[a]	3.240±0.456[a]	2.896±0.096[a]
	Methyl vinyl ketone	20.504±5.672[b]	24.160±7.400[b]	16.656±6.024[a]
	3-Heptanone	0.320±0.024[a]	0.328±0.032[a]	0.320±0.032[a]
	Acetophenone	0.584±0.008[a]	0.624±0.040[a]	0.680±0.024[a]
Acids	Acetic acid	0.248±0.040[a]	0.376±0.064[a]	44.528±7.888[b]
	Benzhydrazide	0.960±0.096[a]	1.024±0.104[a]	0.936±0.056[a]
Aldehydes	Hexanal	0.040±0.000[a]	0.048±0.008[a]	0.048±0.008[a]
	Benzaldehyde	3.224±0.136[a]	3.296±0.040[a]	3.016±0.328[a]

Means values (n = 3) followed by the same letter in the same line are not significantly different ($p > 0.05$); ND: not detected.

Table 4. Concentration (µg/L) of volatile organic compounds identified in palm wine sample collected from local tappers at the seventh day of tapping (P7D) and stored at ambient temperature

Chemical group	Compounds	Storage duration (h)		
		0	24	48
Terpenes	o-Cymene	0.758±0.042[a]	0.895±0.068[a]	0.983±0.084[a]
	beta- Myrcene	0.019±0.003[a]	0.004±0.001[a]	0.004±0.001[a]
	D-Limonene	0.253±0.012[a]	0.424±0.065[a]	0.307±0.064[a]
Alcohols	Isobutanol	46.200±0.846[a]	45.119±9.453[a]	38.243±7.322[a]
	Isoamyl alcohol	231.666±16.597[c]	190.098±9.217[b]	148.815±8.328[a]
	1-Octanol	0.339±0.076[a]	0.024±0.008[b]	0.102±0.091[ab]
Esters	Ethyl acetate	3915.967±50.144[b]	4392.305±90.375[c]	3566.798±72.820[a]
	Methyl butanoate	0.299±0.076[a]	0.343±0.036[a]	0.370±0.056[a]
	Ethyl butanoate	2.610±0.920[a]	3.424±0.371[a]	2.217±0.437[a]
	Orthoformic acid, triisobutyl ester	0.971±0.053[a]	1.481±0.740[a]	1.493±0.768[a]
	Ethyl hexanoate	37.796±4.667[b]	75.676±7.810[c]	24.320±3.098[a]
	Linalyl formate	0.604±0.055[a]	0.950±0.097[a]	0.523±0.062[a]
Ketones	2-Pentanone	3.126±0.260[a]	3.759±0.331[a]	3.542±0.832[a]
	Methyl vinyl ketone	42.815±3.406[b]	38.643±0.568[b]	15.026±0.923[a]
	3-Heptanone	0.334±0.013[a]	0.346±0.041[a]	0.340±0.028[a]
	Acetophenone	0.942±0.038[a]	0.580±0.011[a]	0.631±0.025[a]
Acids	Acetic acid	ND	ND	43.424±6.410
	Benzhydrazide	0.961±0.038[a]	1.282±0.081[a]	1.342±0.482[a]

In order to investigate correlations among volatile profiles of fresh and deteriorating palm wine samples and identify the main components that best discriminate between the samples analysed, a principal components analysis (PCA) was carried out (Figure 2) by considering compounds with significant variations in concentration. Regarding P1D samples, the first two principal components (PCs) of PCA explained 100% of the total variance. The fresh sample (0 h) was clearly separated from 24 h sample on PC1. It was characterized by high concentration of ethyl acetate and the low concentration of isoamyl alcohol, isobutanol and ethyl butanoate. The P1D 48 h sample, was separated from the other samples on PC2 and it was characterized by a high concentration of acetic acid. PC1 (69.99% of variance) and PC2 (30.01% of variance) allowed the separation of P7D samples. P7D 0 h and P7D 24 h were separated by PC2 and P7D 24 h was characterized by a low concentration of isoamyl alcohol and 1 octanol. PC1 separate P7D 48 h from the other samples. This sample was characterized by a high concentration of acetic acid and the low concentration of isobutyl alcohol, isoamyl alcohol, ethyl acetate, ethyl butanoate, ethyl hexanoate and methyl vinyl ketone.

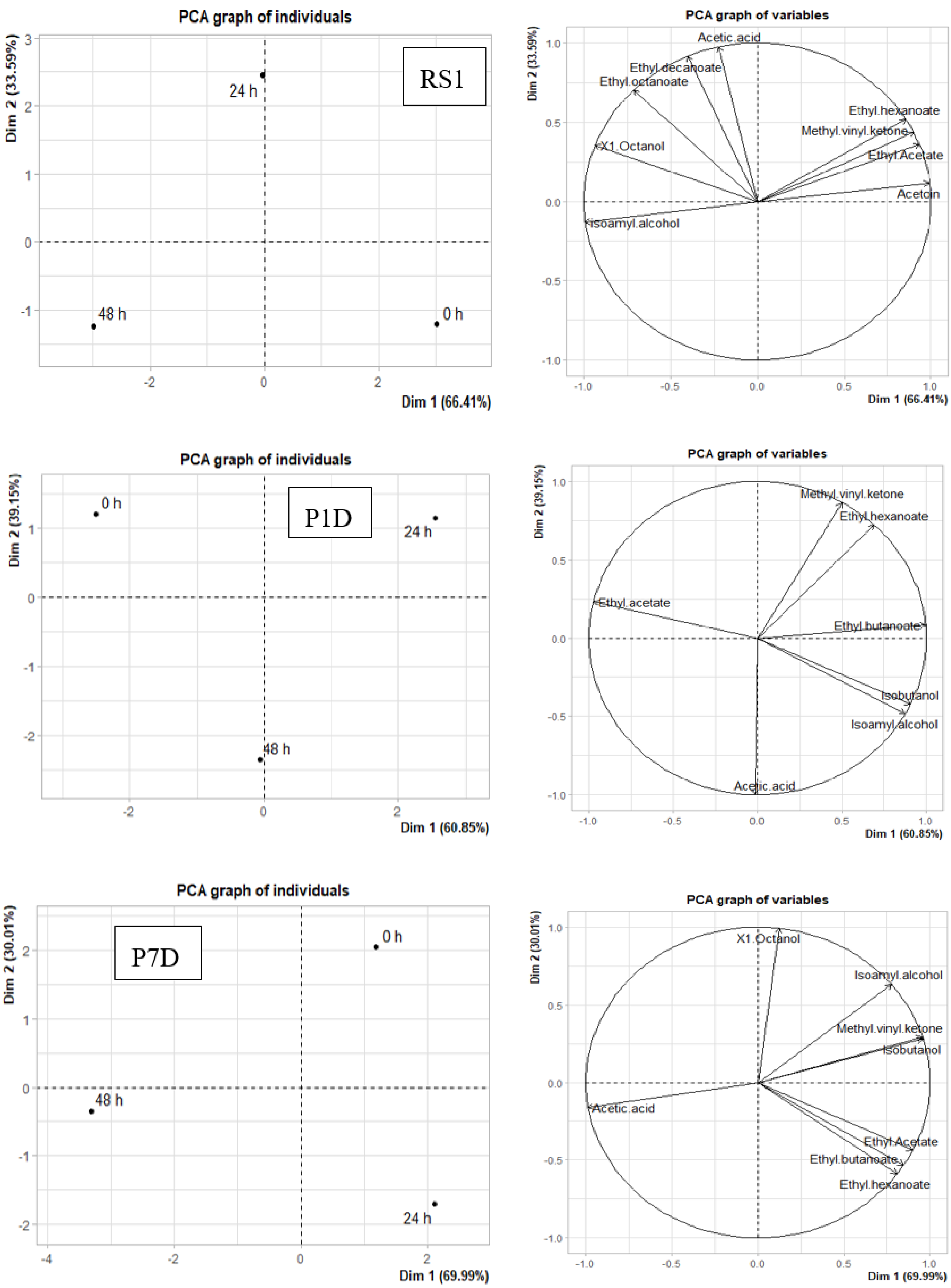

Figure 2. PCA showing the PCs score plots and PC1 and PC2 loading plots obtained from the total chemical families of volatile organic compounds determined in stored palm wine samples
P1D, P7D: palm wine samples collected from the tappers at the day 1 and 7 respectively. RS1: palm wine sample collected from the local reseller 1

Principal Components Analysis of Stored Palm Wine Samples

For RS1 samples, ethyl acetate, ethyl hexanoate, acetoin and methyl vinyl ketone were positively correlated to PC1 which accounted for 66.41% of total variance. PC1 separate RS1 0 h from RS1 48 h which was characterized by a high concentration of isoamyl alcohol and 1-octanol.

Similar to the sensory evaluation, PCA based on total concentration of VOC chemical families revealed differences among samples after 24 h and 48 h, respectively for palm wines from tappers and local resellers. This result is in the same line of those reported by Attchelouwa et al. (2020) for deteriorating sorghum beer samples. These results corroborated those of Sanni et al. (1999) who showed that in traditional beverage like sekete, pito and burukutu spoilage occurred 72 hours after production and was marked by a vinegary flavor and an unpleasant odor.

Conclusion

In this study, the sensory evaluation and volatile compositions were used to monitor the shelf-life of palm wine stored at ambient temperature. Sensory evaluation demonstrated that the rejection time was set at 24 h and 48 h for wines collected from the tappers and local resellers respectively. A total of 23 volatile compounds were identified by the SPME-GC/MS method, which include terpenes, esters, ketones, alcohols, aldehydes and acids. The markers of spoiled palm wine identified were isoamyl alcohol, isobutanol, 1-octanol, aceti acid, acetoin, ethyl acetate and/or ethyl hexanoate. In the view to fight against the palm wine short shelf-life at ambient temperature, the use of bacteria as biopreservatives could be explored.

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Conflicts of Interest

The authors declare no conflict of interest. The funders had no role in the design of the study; in the collection, analyses, or interpretation of data; in the writing of the manuscript, or in the decision to publish the results.

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