



Evolution of physico-chemical parameters, microorganism diversity and volatile organic compound of apple pomace exposed to ambient conditions

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ABSTRACT

In apple processing, waste material known as apple pomace amounts to 45% of production volumes. When this residue is stored in open-air for its stabilization and potential uses, Volatile Organic Compounds (VOCs) are produced, resulting in environmental and odor pollution, and must be managed to avoid their impact. This work aims to study the emission of VOCs utilizing TD-GC/MS and its relationship with changes in physico-chemical (moisture, pH, proteins, among others) and biological (bacteria and fungi using Illumina MiSeq) parameters under three environmental conditions: open-air (outdoors), under-roof (indoors) and oxygen-free. The 8-month study results showed a gradual increase in odorous VOCs and microbial diversity, a product of chemical and biological transformation processes in the samples. A 30% increase in odorant compounds responsible for the unpleasant smell was observed, especially esters, aldehydes and hydrocarbons in samples stored in oxygen-free and Open-air conditions. Increases in VOCs over time were associated with changes in physico-chemical and biological parameters, as well as fluctuations in environmental variables (temperature, relative humidity, and precipitation). The results of this research allow establishing a relationship between storage conditions and the production of VOCs. In addition, recommendations for waste storage time are provided for the most common uses of apple pomace based on the physico-chemical parameters observed, in order to avoid the generation of odorous compounds. Of all storage methods analyzed, under-roof is the most adequate in practice. This study's findings are pertinent for managing agribusiness waste and its potential environmental pollution.

1. Introduction

Apple pomace corresponds to 45% of the apple manufacturing process and consists of remains of pulp (54%), cores (4%), peel (34%) and seeds (7%) [1]. This waste is obtained mainly from the production of apple pulp and juice, and dehydrated apple [2]. In Chile, 1.6 million tons of this residue are produced annually, which is mainly made up of sugars, cellulose, fibers, pectin and antioxidants, among others [3]. The sugars and nutrients of apple pomace have been used as raw material to generate by-products of

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interest such as bioethanol, biopolymer, phenolic antioxidants, citric and lactic acid, enzymes, aromatic compounds, among others [2, 4–6]. However, fresh apple pomace is perishable and highly biodegradable, given its high organic matter content, which on a large scale can cause considerable environmental pollution [4]. It has also been reported through a life cycle analysis that there is an environmental impact of odorous Volatile Organic Compounds (VOCs), when apples are produced, transported, processed and dumped as waste [7]. This situation generates two major environmental problems: a) odor pollution for the surrounding population; and b) damage to plants, surface effluents, groundwater and high eutrophication. For this reason, the correct handling of this by-product, in addition to having the potential to produce monetary income, can reduce the environmental impact and help meet the sustainable development requirements of agribusiness [8].

In Chile, and other countries, apple pomace does not have a specific final destination, becoming a waste that is scarcely used [9].

This waste is often neglected and stored in open-air conditions on agricultural estates or dedicated storage areas. The purpose of storage is to reduce its moisture content and volume so that it can be reprocessed later for different uses, especially bioproducts [2]. The accumulation of apple pomace generates emissions of VOCs especially as a result of natural fermentative processes occurring over time. Microorganisms such as *Saccharomyces cerevisiae*, *S. uvarum*, *Torulaspota delbrueckii*, *Hanseniopsis osmophila*, *H. uvarum*, *Starmerella bacillaris* and *Zygosaccharomyces bailii* have been shown to produce compounds derived from the families of alcohols, esters and fatty acids, such as 2-phenylethanol and hexyl, isoamyl acetate, in apple fermentation processes [10]. Kim et al. [11], showed that ripe apples produce toluene, 1,3-xylene, 3,3-dimethylundecane, butan-1-ol, 2-methylidenebutanal, 6-methylhept-5-en-2-one, 1-2, xylene, α -farnesene, and 2-methylbutan-2-ol when affected by biomass-degrading fungal pathogens such as *P. expansum*, *B. dothidea* and *A. alternata*. This suggests that apple waste, which contains high amounts of carbohydrates, fibers, and polyphenols, will serve as a substrate that would allow microbial growth [5].

The investigation of VOCs emission during the storage and degradation of apple waste holds significant importance, considering the current lack of information on the decomposition mechanisms and the correlation between storage conditions and odorant VOC emissions, microbial diversity changes, and physico-chemical alterations over time. Thus, this study aims to thoroughly examine the temporal evolution of physico-chemical and microbiological characteristics, as well as the generation of VOCs in apple pomace as it undergoes degradation under uncontrolled environmental conditions over 8 months. By conducting this research within three distinct storage conditions, namely open-air, under-roof, and oxygen-free, the influence of environmental factors on the variation of physico-chemical parameters, microbial growth and diversity, and VOC production can be comprehensively analyzed. Properly justifying the necessity of this study lies in addressing the significant knowledge gaps surrounding VOC emissions throughout apple waste storage and degradation, ultimately leading to a better understanding of this complex process and its implications.

2. Materials and methods

2.1. Samples

Apple pomace sample material was collected from SURFRUT company, that processes apples of the Royal Gala, Richared Delicious, Granny Smith, Gala and Tenroy Royal Gala varieties and is located in the commune of Romeral, Chile. Sample material was taken at once from the single process line's waste outlet pipe. Each sample was identified and standardized to a mass of 20 kg and transported and stored in containers for 8 months (winter to spring in the southern hemisphere) under uncontrolled environmental conditions in the laboratory of the University of Talca, Curicó, Chile.

Three apple pomace samples were studied for each storage condition: open-air, under-roof and oxygen-free (9 samples in total). The containers stored outdoors and indoors were kept open to the atmosphere, while the containers oxygen-free were treated with nitrogen and vacuum sealed to remove air. The containers had a gas outlet that was trapped in a Tedlar bag to extract VOC gas samples. Additionally, each container had a lid that was opened once a month to extract solid-liquid samples from within and then it was resealed by applying nitrogen and vacuum sealing. All containers were kept without agitation and movement to simulate what happened in a real process due to the large volumes to be treated.

For the initial characterization, 1 kg of waste sample recently retrieved from the company was used. The samples were analyzed to know the initial characteristics of the residue in triplicate. These measurements were used as a reference for changes that occurred over time in the stored samples. To estimate the degradation of the apple pomace over time on the three storage conditions (open-air, under-roof and oxygen-free), for a period of 8 months, samples were taken monthly from different areas of each container. Each sample consisted of 5 parts of 40 g each, delivering a total of 200 g of sample to be analyzed in each container. For the extraction, a 250 cm² dredge-type sediment sampler (Van Veen, model 12.110, Sidmar) was used. The 600 g obtained in this way for each storage condition were used for the physico-chemical, microbiological and VOCs analyses. All analyzes were performed in triplicate. Additionally, using the same method described above, 600 g of sample were extracted to be stored and frozen at -18°C for later analysis and controls.

2.2. Analytical methods

Physico-chemical analyzes were performed on each sample month by month, using standardized methods (Oven Method—AOAC 945.15, Kjeldahl method—AOAC 979.09, Soxhlet method—AOAC 963.15, Miller method, Gravimetric method—AOAC 920.169, Muffle method—AOAC 940.26, DIN Series 51.900 Standard). For all procedures excluding pH and total phenolic, the samples were dried at $103 \pm 2^{\circ}\text{C}$ using an oven (Mettmert brand model W02WVU), according to the procedures already described in references [12, 13].

The composition of VOCs was determined every two months. For the open-air and under-roof samples, VOCs were extracted

directly from the gas phase of the containers using a suction pump (Markes Easy VOC, LP-1200) to extract 100 ml of the gas phase through adsorption tubes (Markes C2-BAXX-5315 odor/sulfur. C6/7-C30, thiols and mercaptans). In the case of the samples oxygen-free, the gases were extracted from the Tedlar bags, where they accumulated over time. Desorption of the tubes was carried out using a thermal desorption cold trap injector (Markes, Unity-xr), by thermal desorption gas chromatography mass spectrometry (TD-GC/MS) described in references [12,13].

2.3. Reagents

All the standards used corresponded to pure substances from Merck, Sigma-Aldrich and Lancaster Synthesis, with purities greater than 95% and for analysis.

2.4. Environmental conditions

Data on temperature, relative humidity, wind speed and amount of daily precipitations was collected and averaged monthly during the 8 months of the study duration. The information was retrieved from the weather station located at the airport in the city of Curicó [14]. The parameter averages are shown in Table 1. It is important to observe that with the change of season (winter to spring), the minimum and maximum temperature (Tmin and Tmax) increased, whereas the relative humidity decreased. A particular case was that wind and precipitation were variable throughout the experimentation.

2.5. Metal analysis

The metal content in the apple pomace was determined at the beginning and end of the storage process. The equipment used was a Nova 800 Analytik Jena Atomic Absorption Spectrometer (AA), flame, graphite furnace and hydride systems (HS 60). The analysis was carried out on samples of approximately 1 g in three replicates. Samples were combined with 7 ml HNO₃ (65% v/v) and 1 ml H₂O₂ (30% v/v). The samples for the determination of light metal concentrations were diluted to a volume of 25 ml. The modifier for Hg and As elements was 4% NaBH₄. Digestion was carried out under a fume hood, using a hot plate (Labtech Model EH), at a temperature of 60 °C for 8 h. After digestion, the samples were diluted with HPLC grade water to a volume of 50 ml. Metal standards supplied by Merck and minimum analytical grade reagents were used in the analysis.

2.6. Statistical methods

Physico-chemical and environmental variables were analyzed via Pearson correlation, and corresponding plots were obtained using the R package "PerformanceAnalytics" and the estimators provided [15]. All codes necessary to create the plots in this article are available at [16].

2.7. Illumina sequencing analysis

The identification process of the microbial population in the apple pomace samples subjected to the three forms of storage (open-air, under-roof and oxygen-free) was carried out using next-generation sequencing between month 0 (March) and month 8 (October). Each sample was drawn as a mixture from each container. Samples were frozen and sent for sequencing. DNA was extracted from the samples using the MoBio PowerSoil™ DNA extraction kit protocol. The quantity and quality of the extracted DNA were evaluated using a NanoDrop 1000 spectrophotometer (Thermo Fisher Scientific, USA). DNA samples were sent to the Research and Testing Laboratory (RTL, Lubbock, USA). For the sequencing analysis of the bacterial community, the variable V3–V4 of the 16S rRNA was used. In the case of fungi, a set of primers with fungal bias, composed of ITS1/ITS4, was used.

3. Results and discussion

3.1. Temporal evolution of waste physico-chemical parameters and their relationship to environmental factors

Table 2 displays the initial physico-chemical parameters of the samples, which align with values from previous studies. Thus, the samples used in this study accurately represent this type of residue.

Table 1

Average monthly weather conditions.

Meteorological conditions	Units	Mar	Apr	May	Jun	Jul	Aug	Sep	Oct
Precipitation (PPT)	mm	4.0	15.1	45.2	129.4	76.2	60.6	38.2	31.2
Min. Temperature (Tmin)	°C	11.2	9.5	8.3	5.7	4.3	4.7	5.6	7.0
Max. Temperature (Tmax)	°C	28.7	24.1	18.7	14.4	13.2	15.1	18.1	21.1
Wind Speed	Km/h	4.4	9.4	7.5	7.5	9.6	10.5	7.5	4.5
Relative Humidity (RH)	%	47.1	51.2	60.2	67.6	72.3	73.6	67.5	63.4

Table 3 shows the evolution of the parameters analyzed throughout the study period. The data is separated by each environmental condition studied: open-air, under-roof and oxygen-free. Physico-chemical properties included in the analysis correspond to the mean values of ash, calorific value, carbohydrate, fat, fiber, moisture content, pH, protein and total phenolic content samples performed in triplicate. The last column details the percentage of relative variation between the maximum and minimum data for each parameter throughout the study period. Table 3 illustrates the degradation and increase of compounds of the parameters considered according to the storage condition. In the case of the samples under oxygen-free conditions, they were kept at room temperature, being affected by the Tmin and Tmax, but without being affected by the wind and relative humidity.

A Pearson correlation analysis was done between most relevant physico-chemical and environmental variables. Fig. 1 shows the correlation values between physico-chemical parameters and environmental variables. Correlations with significant *P*-values ($P < 0.05$) are marked with a "*". To see the distributions and levels of significance of all the variables, please refer to the supplementary material at the end of this document.

Tmax, Tmin and RH were the environmental parameters that presented the most significant statistical correlation between them and with the physico-chemical parameters. In the case of precipitations and wind, they didn't present significant *p* values, but they affected the samples conditions, especially in winter in open-air conditions and parameters such as moisture content that presented moderate correlation values (0.61).

The moisture content of the open-air samples increased with precipitations and relative humidity as expected from the environmental data presented in Table 1. In the rainiest month (June) the moisture content increased up to 96.8%, this is 12.7% more than at the beginning. During the storage period, the Tmin and Tmax also decreased, which decreased the water evaporation. However, for the under-roof samples, moisture content continued to decrease by 69.1% due to evaporation. For samples under oxygen-free conditions, the moisture content increased by 11.9% due to its natural decomposition process, but without the possibility from evaporating when stored in a completely closed tank. Correlation values show that there is a strong positive correlation (0.78) between moisture content and relative humidity ($P = 0.0226$) as it is a well-known phenomenon that internal RH tends to increase to the external RH levels. The ability to release water from apple pomace under various temperature conditions is known. A study developed by Ref. [27] shows that the dehydration of apple pomace at 40 °C is achieved in 24 h or more, reaching humidity values of 6% w/w.

Proteins underwent changes in storage time that can be attributed to environmental conditions. In the open-air and under-roof samples, there was an increase of 19.3% and 37.3% in the months of March and April compared to the beginning, and then remained relatively stable over time. In the case of the samples in oxygen-free conditions, the behavior over time was different; the maximum level of proteins was reached in the month of July (25.3%), that is, after five months and the change percentages in proteins throughout the period were highest, reaching a maximum of 104.2%. During March and April, the Tmin and Tmax were the highest in the study period and there were no precipitations. This leads us to assume that temperatures and precipitations affected the percentage of proteins during waste storage, especially helping hydrolysis processes to occur [28]. these results in the case of oxygen-free condition have a significant statistical relevance as there is a negative correlation value with Tmin (−0.77) and Tmax (−0.89). A study carried out by Ref. [17] shows that in apple pomace the protein content increases in the first 48 h and then slowly decreases over time as a result of the natural fermentative processes that occur and increase the microbial mass. This is due to the increase in *Aspergillus niger* that produces cellulase. In the production of bioethanol from apple pomace [2], it was also found that by fermentation the proteins increase from 1.3% to 8.2%.

Fats and fibers showed similar behaviors, as they tend to decrease in under-roof and anaerobic samples. Fats of under-roof and anaerobic samples decreased by −22.9% and −16.6%, respectively, while in open-air samples, fats remained relatively stable with a difference of only −2.9%. On the other hand, fibers registered a change of −8.6%, −31.9% and −62.4% for open-air, under-roof and

Table 2

Parameters of apple pomace samples before storage period. Standard deviation values are shown in parentheses.

Properties	Unit	Determination Method	This study	Literature range	References
Moisture content ^a	Mass %	Oven method—AOAC 945.15	85.5 (1.1)	67.92–86.90	[2] [17]
Proteins ^b	Mass %	Kjeldahl method—AOAC 979.09	7.6 (0.07)	1.35–64	[2] [18]
Fats ^b	Mass %	Soxhlet method—AOAC 963.15	4.6 (0.1)	0.71–2.2	[2] [19]
Carbohydrates ^b	Mass %	[20]	15.4 (0.89)	10.36–18.3	[2] [21]
Fiber ^b	Mass %	Gravimetric method—AOAC 920.169	52.4 (1.01)	36.8–74.69	[22] [23]
Ashes ^b	Mass %	Muffle method—AOAC 940.26	2.7 (0.11)	0.65–4.7	[2] [24]
Calorific value ^b	MJ/kg	DIN Serie 51.900 Standard	18 (0.03)	18.7–2232	[2] [25]
pH (juice) ^a	DI		4 (0.04)	3.8–4.5	[2] [21]
Total phenolic content ^a	mg/g	Folin-Ciocalteu's method as gallic acid equivalents	3 (0.02)	1.5–8.9	[19] [26]

^a Wet basis.

^b Dry basis.

Table 3
Evolution of physico-chemical parameters of apple pomace samples. Standard deviation values are shown in parenthesis.

	Property	Unit	Mar	Apr	May	Jun	Jul	Aug	Sep	Oct	% Change
Open-air	Ashes	Mass	3.5	3.2	3.1	3 (1.17)	2.9	2.1 (1.1)	2.7	2.2	-37.0
		%	(0.98)	(0.94)	(0.98)		(1.03)		(0.01)	(0.11)	
	Calorific value	MJ/kg	18 (1)	18	18.4	13.7	21.3	24.8	23.2 (1)	22.3	23.7
		%		(1.04)	(0.25)	(10.2)	(0.38)	(0.94)		(1.25)	
	Carbohydrates	Mass	15.5	18.2	35	35	35.9	54.9	55.8	59.5	282.7
		%	(2.02)	(0.95)	(1.42)	(1.01)	(0.95)	(0.13)	(1.05)	(0.48)	
	Fats	Mass	4.3	4.3	4.9	4.9	4.9	4 (0.19)	4 (0.24)	4.2	-2.9
		%	(0.99)	(0.83)	(0.13)	(0.58)	(0.21)			(0.21)	
	Fiber	Mass	53.2	54.2	53.1	53.5	52.2	51.2	50.1 (1)	48.6	-8.6
		%	(1.01)	(0.16)	(0.99)	(1.84)	(1.12)	(1.1)		(1.01)	
Moisture content	Mass	84.5	85.6	92.5	96.8	95.8	94.2	70.1	60.4	-28.5	
	%	(1.23)	(1.83)	(1.14)	(1.58)	(2.53)	(4.29)	(2.94)	(2.45)		
pH (juice)	DI	4 (0.99)	3.9	3.3	3.5	3.7	3.8	3.2	3.1	-22.2	
			(0.01)	(0.81)	(0.33)	(0.98)	(0.05)	(0.09)	(0.02)		
Proteins	Mass	8.4 (1)	10.4	7.4	8 (0.93)	7.6	7.6	6.4	5.1	-39.2	
	%		(0.9)	(1.08)		(0.95)	(1.55)	(0.99)	(1.01)		
Total phenolic content	mg/g	2.9 (1)	2.8	2.7	2.5	2.3	2.2	2.2	2 (0.04)	-31.2	
	%		(0.05)	(0.99)	(0.16)	(0.16)	(0.13)	(0.72)			
Under-roof	Ashes	Mass	2.8 (0.9)	3.3	3.7	3.3	3.4	3.8	3.6 (0.1)	3.9	37.0
		%		(0.62)	(0.89)	(0.62)	(0.88)	(0.94)		(0.05)	
	Calorific value	MJ/kg	18.3	18.4 (1)	18.7	19.5	20.9	21.5	22	22.3	21.6
		%	(0.16)		(0.73)	(0.57)	(0.95)	(1.22)	(0.98)	(1.23)	
	Carbohydrates	Mass	15.9	20.3	30.3	34.6	36.1	37.7	39.8	40.4	154.1
		%	(0.81)	(2.04)	(1.39)	(1.02)	(0.3)	(1.06)	(1.79)	(0.2)	
	Fats	Mass	4.6	4.4 (1)	4.5	4.5	3.7	3.7	3.8	3.5	-22.9
		%	(0.06)		(1.12)	(0.01)	(0.85)	(0.16)	(1.06)	(0.03)	
	Fiber	Mass	52.2	50.3	52.3	53.5 (1)	48.4	44.6	44.3	35.5	-31.9
		%	(0.98)	(2.25)	(0.86)		(2.94)	(0.09)	(1.67)	(0.16)	
Moisture content	Mass	85.3	82.5	81	79.9	74.7	61.1	40.4	26.3	-69.1	
	%	(0.96)	(4.13)	(4.07)	(3.51)	(2.26)	(2.46)	(3.05)	(2.17)		
pH (juice)	DI	4 (0.06)	3.7 (0.2)	3.4	3.8	3.4	3.8	3.7 (0.2)	3.2	-20.0	
				(0.26)	(0.09)	(0.05)	(0.09)		(0.09)		
Proteins	Mass	8.4	13.4	8.1 (1.2)	8.1	8.6	8.5	7.4	7.1 (2)	-15.5	
	%	(1.09)	(2.06)		(1.11)	(0.84)	(0.17)	(1.05)			
Total phenolic content	mg/g	3.1	3.3 (0.2)	3.2	2.8	2.2	2.3	2.1	1.7	-45.0	
	%	(0.33)		(0.23)	(0.87)	(0.22)	(0.01)	(0.19)	(0.19)		
Oxygen-free	Ashes	Mass	3.3	3.6	3.9	4 (0.01)	4.5 (0.1)	4.1	4.3 (0.5)	4.1	23.2
		%	(0.09)	(1.21)	(0.01)			(0.11)		(0.09)	
	Calorific value	MJ/kg	19.7	21	19.5	21.7	20.5	21.5	22.3 (0)	22.7	15.3
		%	(0.94)	(0.02)	(1.33)	(1.14)	(1.22)	(1.27)		(0.1)	
	Carbohydrates	Mass	17.6 (0)	12.8 (1)	60.8 (1)	80.5	90.6	85.5	82.4	80.1	353.8
		%			(1.04)	(0.66)	(0.27)	(1.16)	(1.01)		
	Fats	Mass	4.9	5 (1.01)	5.1 (0.8)	5 (0)	4.9	4.8 (0)	4.5	4.1	-16.6
		%	(1.05)				(0.42)		(0.31)	(0.13)	
	Fiber	Mass	52.2	53.2	52.1 (1)	51.5	43.4	32.2 (1)	21.2	19.6	-62.4
		%	(1.01)	(0.22)		(0.05)	(0.43)		(0.09)	(0.95)	
Moisture content	Mass	85.6	86.5	87.1	89.4	93.7	93.7	94.8 (1)	95.8 (1)	11.9	
	%	(1.01)	(1.05)	(1.43)	(1.06)	(1.11)	(0.5)				
pH (juice)	DI	3.9	2.2	3.7	3.7	3.7	3.7	3.4 (0)	3.2	-18.5	
		(0.01)	(0.01)	(0.01)	(0.11)	(0.02)	(0.05)		(0.04)		
Proteins	Mass	7.4	12.8	18.6	20.3	25.3	15.5	15.6	15.2	104.2	
	%	(0.98)	(0.99)	(0.02)	(0.07)	(0.23)	(1.15)	(0.27)	(0.98)		
Total phenolic content	mg/g	2.7	2.4	2.6	1.6	1.6	1.2	1.2	1.1 (0)	-59.4	
	%	(0.01)	(0.11)	(0.03)	(0.11)	(0.11)	(0.07)	(0.01)			

oxygen-free storage conditions, respectively. Ambient temperatures (T_{min} and T_{max}) can directly affect fiber and fat degradation over time, as reported by Ref. [29] for apple pomace flour.

In the case of proteins, fats, and fibers, these parameters are relevant because they allow determining whether a product exhibits significant physico-chemical, and biological properties to be used for dietary or energy purposes.

Ashes had different behaviors depending on the type of storage. In both under-roof and oxygen-free samples, ashes increased rapidly during the first months of storage. During the study period, these samples had an increase of 37.0% and 23.2%, respectively. This can be explained as a result of the biological and chemical decomposition processes of the organic matter into CO_2 and VOCs, generating greater leaching of the inorganic matter. Therefore, as the samples were taken every month in the same mass amount, this parameter increased. In open-air samples, ash percentages fluctuated down by -37.0%. In these samples, ash content decreased because samples were taken every month from the same container, which being exposed to rainfall and ambient humidity resulted in

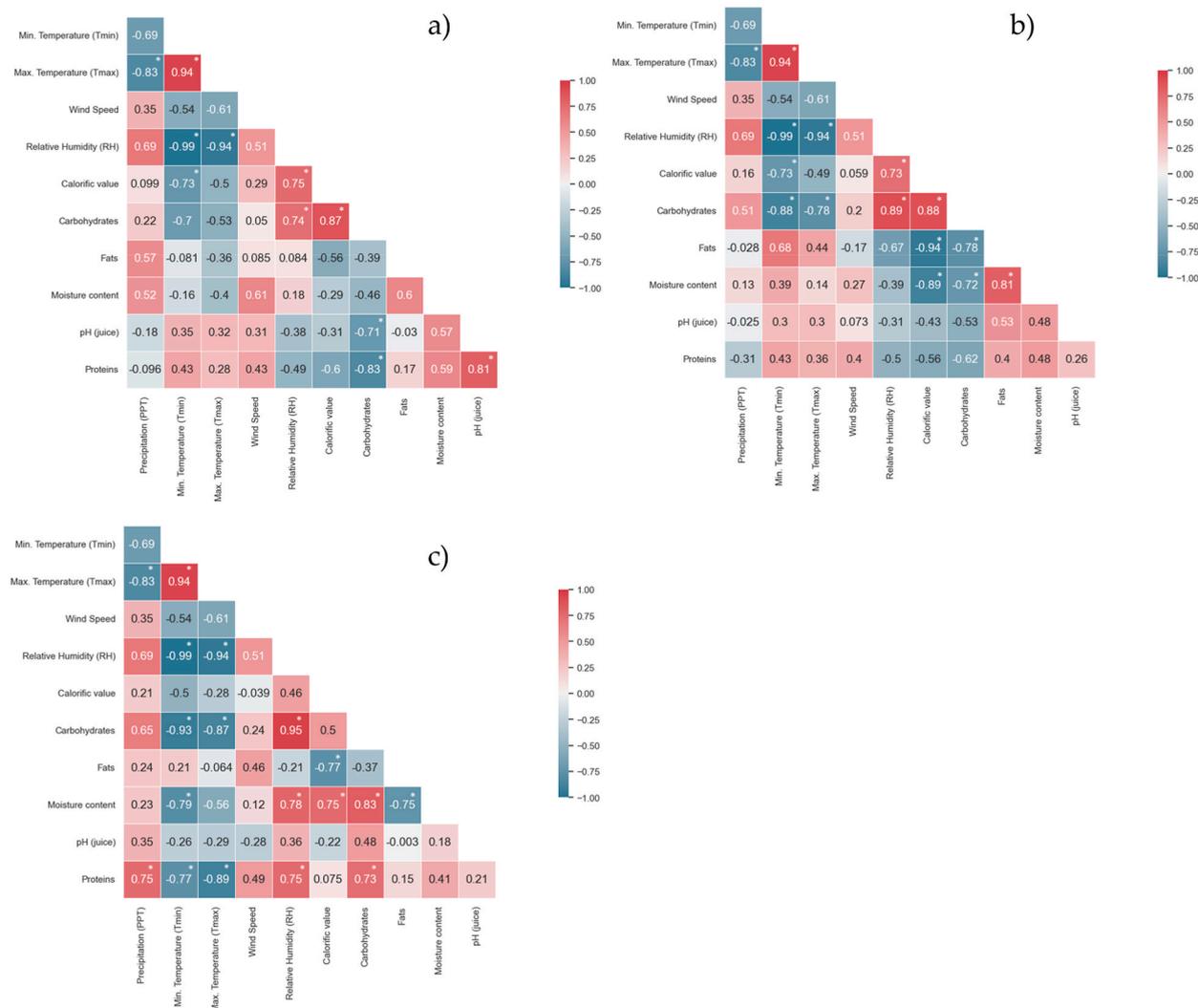


Fig. 1. Correlation matrix of the main physico-chemical properties and environmental conditions for the three storage methods. a) Open-air; b) Under-roof; c) Oxygen-free. Values marked with a * present a P value < 0.05.

dilution and therefore less chemical and biological degradation. Similar results have been reported by Ref. [30], where under certain temperature conditions, the ash content of apple pomace in aqueous medium increases first and then decreases, which is the product of organic matter separation.

Carbohydrates increased by 282.7%, 154.1%, and 353.8% in the open-air, under-roof and anaerobic conditions, respectively. This process may be associated with the pH that decreased for all samples by -20.4% (under-roof), -22.2% (open-air) and -18.5% (oxygen-free). In open-air conditions there is a negative correlation (-0.71) between carbohydrates and pH (P = 0.048). This may be the product of the hydrolysis process that occurs in an acid medium, as reported for apple pomace samples [31].

The total phenolics decreased by -45.0%, -31.2% and -59.4% for the open-air, under-roof and anaerobic samples. These data are directly consistent with the decrease in Tmin and Tmax. Similar results were reported by Ref. [32] for thermally degraded apple pomace flour samples.

The caloric value of under-roof storage, open-air storage and anaerobic storage samples increased by 21.6%, 23.7% and 15.3% respectively. Caloric value presented high correlations with carbohydrates in all conditions (0.87, 0.87) for open-air and under-roof storages, with significant p values (0.0047, 0.0049) for the same conditions. This is primarily attributed to a significant increase in carbohydrate concentrations observed during the study period, indicating a higher amount of energy available for consumption. These results are interesting because the environmental conditions do not affect the caloric value, rather they favor it. Recent studies have shown that apple pomace mixed with other waste can be used for pellet production [33]. This shows that these residues, even after several months of storage under different environmental conditions, can be successfully used for pellet production.

The results reported here demonstrate a link between changes in physico-chemical parameters and the environmental conditions under which the sample experiments are performed. This allows us to determine that apple pomace waste cannot be stored in any way if it is expected to maintain certain properties over time.

3.2. Time evolution of VOCs

VOCs are low molecular weight molecules that become gaseous under certain pressure and temperature environmental conditions [34].

Fig. 2 shows the main VOCs identified throughout the eight months of research, But more than 200 compounds were identified in the samples analyzed. Many of these compounds presented very low concentrations, below odor thresholds described in the literature and there is a significant number of compounds that are repeated over time. Families of hydrocarbons, alcohols, and esters emerged in all samples. These are synthesized from C16:0, C18:0, C18:1, C18:2 and C18:3 fatty acids, a product of amino acid and carbohydrate metabolism derived from synthesis such as β -oxidation, lipoxigenase, isoleucine among others [35] and in this study they were produced even under open-air storage conditions and despite precipitations. All compounds found agree with the NIST library of TD-GC/MS equipment with less than 98% reliability.

The heavier odoriferous compounds found correspond to the families of aldehydes, esters, and hydrocarbons. Of all the VOCs responsible for the unpleasant odor released into the environment, Aldehydes such as hexanal present the highest concentrations in the samples under anaerobic conditions and the lowest in the under-roof samples, as shown in Fig. 2. These unpleasant odor results are similar to those reported by Ref. [36] for hexanal, regarding an analysis of VOCs in salmon when the cold chain is interrupted.

As for esters, a similar trend is observed regarding their emergence between samples stored indoors and outdoors, although more esters are found in under-roof samples. In particular, differences are observed in ethyl butanoate and ethyl propionate. Under-roof samples show a delay in the emergence of ethyl butanoate, while ethyl propanoate shows a dilution at the end of August and tends to disappear. In the samples oxygen-free, there is an increase in almost all volatile ester compounds, which can be associated with the generation of very unpleasant odors by these stored samples.

Regarding hydrocarbons, there is a lower generation of α -farnesene when leaving the waste stored under-roof. In this environment, the α -farnesene compound decreases its concentration from August, the same for 2-methyl pentane. On the other hand, the emergence of 3-methyl pentane in samples stored under-roof delays its emergence compared to open-air samples. Similar measurements are recorded for pentane and n-octane in open-air and under-roof samples.

The results suggest that keeping waste indoors reduces esters, which represent the most aggressive VOCs.

The results obtained are consistent with those presented in previous studies that show the presence of VOCs emitted by apple pomace. Kim et al. [11] demonstrated the presence of about 130 VOCs from families of alcohol, esters, aldehydes, ketones, ethers, and hydrocarbons generated during the decomposition of the apple by different types of fungi such as *Penicillium expansum*, *Botryosphaeria dothidea* and *Alternaria alternata*. In other studies carried out by Ref. [37], the presence of 132 VOCs is demonstrated when apple pomace undergoes fermentation processes for 21 days using different yeasts (*Saccharomyces cerevisiae*, *Hanseniaspora valbyensis* and *Hanseniaspora uvarum*).

Additionally, Fig. 2 shows the limit concentrations of odor threshold (in ppm) reported by different authors [38–41], demonstrating that eight of the compounds found exceed these values. Esters are the compounds that appear the most in this study and are the main aromas of apples, especially 2-methylbutyl acetate [42], which is found in abundant concentration throughout the study period in the 3 samples.

Family	VOC	Odor Threshold	Open Air					Under Roof					Oxygen Free				
			Mar	Jun	Aug	Oct	% Change	Mar	Jun	Aug	Oct	% Change	Mar	Jun	Aug	Oct	% Change
Hydrocarbon	n-pentane	1.4	0.09	1.14	2.23	2.34	2500	3.09	0.98	1.45	1.98	-36					
	n-octane	1.7	0.06	1.16	1.67	2.76	4500	0.01	0.12	0.23	1.45	14400			1.2	1.9	58
	pentane, 2-methyl	7	1.29	0.34			-74	1.21	0.02			-98					
	Pentane, 3-methyl	8.9		0.45	1.45	1.89	320			1.33	1.45	9					
	2-methyl-1-butanol	0.003			1.1	1.5	36				1.4						
Alcohol	Butan-1-ol	0.005	0.91	1.12	1.23	1.98	118	0.21	0.67	0.78	1.21	476	0.98	1.12			14
	Ethanol	0.52	0.02	1.45	0.23		1050	1.12	0.34			-70	0.34	1.14			235
Carboxylic Acid	Acetic acid	0.006	0.23	0.67	1.34	3.43	1391	0.21	0.54	0.76	1.23	486					
Aldehyde	Hexanal	0.005											0.45	1.23	2.34		420
	Acetaldehyde	0.0015			0.98	1.23	26		0.05	0.34	0.78	1460			0.89	2.45	175
Ester	Methyl isovalerate	0.002												0.45	1.32		193
	Methyl butanoate	0.0076			0.56	1.32	136			0.21	0.34	62	0.23	0.98	1.65		617
	Ethyl butanoate	0.001		0.12	0.78	1.12	833			0.23	0.67	191		0.87	0.34		-61
	Ethyl propanoate	0.01		0.23	0.98	1.23	435		0.15	0.23		53	0.56				0
	Ethyl butyrate	0.0018	0.11	0.13	0.14	0.24	118	0.12	0.13	0.14	0.16	33	0.45	0.67	0.12		-73
	2-methylbutyl acetate	0.313	0.02	0.12	1.45	2.49	12350	0.01	0.09	1.23	1.98	19700	0.09	1.11	1.67	2.45	2622

Fig. 2. Main COVs identified in apple pomace samples stored under three environmental conditions for 8 months (samples taken in March, June, August and October). Concentrations are displayed in ppm and colored when surpassing known values of odor threshold.

Other Esters that increased their concentration in the final months in the samples are methyl isovalerate, methyl butanoate, ethyl butanoate, ethyl propanoate, ethyl acetate and ethyl butyrate, which are representative of the aromas that exist when apples decompose [11], as a consequence of the catabolism of fats and acids [43].

Alcohol was another substance that emerged in abundance in the first few months of experiments under the three storage conditions but was higher in those placed indoors and outdoors. Among the most prominent are 2-methyl-1-butanol, cyclohexan-1,4,5-triol-3-ol, hexanol, octanol, butan-1-ol and especially ethanol. These alcohols are generated as a consequence of the decomposition process [37] that produces the reduction of aldehydes by the action of the enzyme alcohol dehydrogenase and the catabolism of acids and fats together with pH changes (see Table 3) [35,44].

Aldehydes were also present in the three conditions, but with a greater presence from August onwards. These compounds are often responsible for the development of pleasant and unpleasant aromas due to the decomposition of chloroplasts as a result of the tissue breakdown and the release of linoleic and linolenic acids through the lipoxygenase pathway [45].

Hydrocarbons such as n-octane, n-pentane, pentane, 2-methyl, 3-methyl were also present in significant quantities in all samples up to October. Previous studies had already reported abundance of hydrocarbons, such as pentane and α farnesene in apple pomace [46], which confirms the results obtained in our study.

Oxygen-free samples produced a smaller number of VOCs than the samples exposed to the open-air. Special attention is paid to Hexanal and Methyl isovalerate, which were only present in this medium. When observing the molecules of Pentane, 3-methyl, Acetaldehyde, Methyl butanoate, Ethyl butanoate and Ethyl butyrate, higher concentrations can be observed in the anaerobic environment (samples oxygen-free) than in the aerobic environment (under-roof and open-air samples). This is especially related to the fact that VOCs are more concentrated when samples are in a closed environment (oxygen-free) than in an open environment, influenced by environmental conditions such as temperature, relative humidity and wind (Table 2). These results are similar to those reported by Ref. [47], which show the vertical distribution of VOCs influenced by environmental conditions. It should be emphasized that the VOCs produced in an anaerobic environment were mainly aldehydes, and their concentrations were higher than the odor thresholds reported in the literature. These results are similar to those reported by Ref. [48], regarding VOCs produced in an anaerobic environment for the stability of biosolids, product of the action of diverse microbial communities and oxidative abiotic transformations.

The results show that VOC emissions varied by molecular type and family during the 8-month storage period under the three storage conditions studied. Special attention must be paid to esters, aldehydes and hydrocarbons which have an unpleasant odor to the environment, especially when they are stored indoors or outdoors in the open-air. This result is directly related to environmental factors, such as temperature as reported by Ref. [49] in a study about the effect of temperature on the distribution of VOCs in the urban area.

3.3. Apple pomace metals

Table 4 shows the initial (month 1) and final (month 8) results of the metals found in the studied samples. The results show changes in the concentrations with average differences of 5% with respect to the starting month. In the three storage conditions studied, the concentrations of Ca and Mg decreased -0.1% and -12.5% (open-air), -7.9% and -9.5% (under-roof) and -7.2% and -15.8% (oxygen-free), respectively. Ca and Mg are elements that regulate pH and are relevant in the fermentation and production of apple pomace biogas. They should not exceed 2800 mg/dm^3 and 4800 m/dm^3 , respectively, in order not to impede the formation of methane [50].

Cu, Fe, Mn and Zn increased their concentrations for all samples 17.1% , 20.9% , 37.3% and 29.2% (open-air), 2.9% , 20.8% , 32.0% and 23.7% (under-roof) and 11.0% , 47.7% , 32.9% and 38.8% (oxygen-free), respectively. The previously described results for Cu, Zn, Mn and Fe are within the reported values of 2.3, 8.91, 5.49 and 33.3 mg/kg , respectively, for apple pomace used as a source of biomolecules [27,51].

The increase in the concentration of metals may be related to the presence of the functional groups $-\text{COO}$, $-\text{CO}$, $-\text{NH}_2$ and $-\text{OH}$ associated with the presence of polyphenols in apple pomace which, according to Chand et al. [47], are responsible for the metal-binding capacity.

Table 4

Metals found at the beginning and end of the study period (8 months), expressed in ppm (mg/kg) for apple pomace samples. AA Spectrometer Method. Standard deviation values are shown in parentheses.

Name	Open-air			Under-roof			Oxygen-free		
	Initial	Finish	% Change	Initial	Finish	% Change	Initial	Finish	% Change
Ca	3702.93 (2.94)	3698.65 (1.99)	-0.1	3724.21 (7.99)	3428.11 (1)	-7.9	3701.12 (11)	3434.11 (2)	-7.2
Mg	112.81 (0.5)	98.62 (1.01)	-12.5	113.52 (0.99)	102.68 (7.54)	-9.5	112.4 (1)	93.58 (0.98)	-15.8
Cd	<0.5	<0.5	-	<0.5	<0.5	-	<0.5	<0.5	-
Pb	<0.5	<0.5	-	<0.5	<0.5	-	<0.5	<0.5	-
Cu	3.95 (0.02)	8.77 (0.21)	17.1	3.8 (0.09)	5.55 (0.03)	2.9	3.88 (0.01)	6.36 (0.01)	11.0
Fe	0.49 (0.01)	0.62 (0.01)	20.9	0.37 (0)	0.65 (0.01)	20.8	0.35 (0.02)	0.67 (0.01)	47.7
Mn	0.57 (0)	0.91 (0.01)	37.3	0.55 (0)	1.15 (0)	32.0	0.52 (0.01)	0.79 (0.02)	32.9
Zn	1.5 (0.1)	3.12 (0.03)	29.2	1.54 (0.1)	2.41 (0.24)	23.7	1.51 (0.1)	3.46 (0)	38.8
Hg	<0.5	<0.5	-	<0.5	<0.5	-	<0.5	<0.5	-
As	<0.5	<0.5	-	<0.5	<0.5	-	<0.5	<0.5	-

Of the 3 storage conditions studied, those that were “under-roof” presented less change in metal concentrations. These results are consistent with the variation of environmental factors in Table 1, because in the case of open-air samples, they were more affected by temperature and precipitation. While in the case of the samples oxygen-free, they were only affected by the temperatures, but for both cases (oxygen-free and outdoors), the solid became more liquid (see Table 3), generating subsequently a greater leaching of metals as a result of biological activity and pH change. A study carried out by Ref. [52] shows that Zinc increases in urban soils treated with compost, due to pH change, the availability of adsorption sites and the microbial activity of the soil. On the other hand, a study carried out by Ref. [53] regarding the behavior of metals in a compost of mixed waste, it is observed that metals increase their concentration over time, as a result of the mineralization of the organic matter and the loss of mass by the various chemical and biological transformations over time.

3.4. Microbial community

This section presents the results of the study on the microbial communities in the samples. The objective is to evaluate the possible influence of biological fermentation processes on waste degradation and the production of odorant VOCs.

Table 5 shows the most abundant fungal found in the samples in months 1 and 8. Initially, the three treatments present a greater abundance of members of the phylum *Ascomycota* with values of 88.9%, followed by the phylum *Basidiomycota*. Special attention is given to the genus *Cladosporium*, *Monilinia*, *Didymella*, *Alternaria*, *Pichia*, *Candida*, and *Aspergillus*, which decrease their abundance over time by 27.70% for open-air samples, 35.91% for under-roof samples and 32.52% for samples oxygen-free. Genus *Cladosporium* and *Alternaria* are responsible for the browning process of Fuji apples [54]. On the other hand, an increase of 10.9%, 13.5% and 0.8% in the genus *Aureobasidium* and *Filobasidium* over time can be observed under open-air, under-roof and oxygen-free conditions, respectively. Similar results were reported by Abdelfattah et al. [51] in a study of microbial diversity in Royal Gala apples. The presence of *Candida* was also found, which has been reported in the bioconversion process of apple pomace as a nutritionally enriched substrate [55].

Table 6 shows the most abundant bacteria for the three samples during the 8-month storage period. The most abundant phylum was *Pseudomonas* at 70%, followed by *Proteobacteria* at 20% and *Bacteroidetes* at 10%. The bacteria of the genus *Acinetobacter*, *Cronobacter*, *Acidiphilium*, *Citrobacter*, *Enterobacter*, *Gluconobacter* and *Acetobacter* increased by 59.2%, while those of the genus *Hymenobacter* and *Erwinia* decreased by 7.9%. In the case of under-roof samples, the genus *Acinetobacter*, *Cronobacter*, *Acidiphilium*, *Hymenobacter*, *Citrobacter*, *Enterobacter*, *Gluconobacter* and *Acetobacter* increased by 62.6%, while the genus *Erwinia* decreased by 11.3%. For the samples oxygen-free, the genus *Enterobacter*, *Gluconobacter* and *Acetobacter* increased by 26.8%, while those of the genus *Acinetobacter*, *Cronobacter*, *Hymenobacter*, *Erwinia*, *Acidiphilium* and *Citrobacter* decreased by 20.2%.

When comparing the growth of the microbial communities of fungi and bacteria shown in Tables 5 and 6, with other parameters measured in this study, a direct relationship can be observed with the VOCs emitted by the samples during the storage process described in Fig. 2 and microbial growth. A study carried out by Ref. [56] shows that the phylum *Ascomycota* and *Basidiomycota* are important for the production of VOCs when they act in an organic medium, especially producing compounds from the families of alcohols, hydrocarbons and carboxylic acids. On the other hand, a study carried out by Ref. [57] shows that there is an increase in VOC emissions (hydrocarbons, aldehydes, ketones, among others) in soils from organic waste when phylum *Proteobacteria* diversities are present.

As can be seen in Tables 5 and 6, the fungal and bacterial communities changed significantly over storage time of the waste. However, these changes were the product of the environmental conditions reported in Table 1, resulting in changes in pH and C/N ratios in the samples. These results are similar to those reported by Ref. [58], where they evaluate the effect of the physico-chemical properties on fungi and bacteria developing in composted samples of agricultural waste. On the other hand [59], shows that in samples of organic matter extracted from water for composting, fungal and bacterial communities change over time, based on the humidification process.

As some of the most common ways to reuse apple pomace include composting, pelletizing, alternative feedstuffs, and biogas [60], it is important to summarize how the pertinent parameters evolve in time and in what storage conditions.

Table 5
Most abundant fungal OTUs found in O and A with relative abundance higher than 5%.

Phylum	Class	Order	Genus	Relative fungal abundances in apple pomace %					
				Open-air		Under-roof		Oxygen-free	
				Month 1	Month 8	Month 1	Month 8	Month 1	Month 8
<i>Ascomycota</i>	<i>Dothideomycetes</i>	<i>Capnodiales</i>	<i>Cladosporium</i>	8.5	5.4	10.4	2.1	4.5	0.5
<i>Ascomycota</i>	<i>Leotiomycetes</i>	<i>Helotiales</i>	<i>Monilinia</i>	3.5	2.5	3.9	0.6	4.9	1.7
<i>Ascomycota</i>	<i>Dothideomycetes</i>	<i>Pleosporales</i>	<i>Didymella</i>	3.5	1.2	5.3	0.1	3.7	1.2
<i>Ascomycota</i>	<i>Dothideomycetes</i>	<i>Dothideales</i>	<i>Aureobasidium</i>	0.5	1.6	0.3	2.5	0.5	0.9
<i>Ascomycota</i>	<i>Pleosporaceae</i>	<i>Pleosporales</i>	<i>Alternaria</i>	10.5	0.5	12.4	0.1	11.4	0.2
<i>Basidiomycota</i>	<i>Tremellomycetes</i>	<i>Filobasidiales</i>	<i>Filobasidium</i>	0.5	10.4	1.2	12.5	0.5	0.9
<i>Ascomycota</i>	<i>Saccharomycetes</i>	<i>Saccharomycetales</i>	<i>Pichia</i>	1.2	0.9	2.3	1.5	1.3	0.3
<i>Ascomycota</i>	<i>Saccharomycetes</i>	<i>Saccharomycetales</i>	<i>Candida</i>	12.2	1.2	12.3	6.3	11.2	0.9
<i>Ascomycota</i>	<i>Eurotiomycetes</i>	<i>Eurotiales</i>	<i>Aspergillus</i>	1.12	0.22	1.11	0.12	1.34	0.02

Table 6

The most abundant bacterial OTUs found in apple pomace with relative abundance higher than 5%.

Phylum	Class	Order	Genus	Relative bacterial abundances in apple pomace %					
				Open-air		Under-roof		Oxygen-free	
				Month 1	Month 8	Month 1	Month 8	Month 1	Month 8
<i>Pseudomonadota</i>	<i>Gammaproteobacteria</i>	<i>Pseudomonadales</i>	<i>Acinetobacter</i>	1.3	1.7	1.5	2.4	2.8	0.9
<i>Pseudomonadota</i>	<i>Gammaproteobacteria</i>	<i>Enterobacterales</i>	<i>Cronobacter</i>	3.5	23.3	2.6	28.6	3.7	0.7
<i>Bacteroidete</i>	<i>Cytophagia</i>	<i>Cytophagales</i>	<i>Hymenobacter</i>	1.3	0.7	0.9	1.3	0.9	0.1
<i>Pseudomonadota</i>	<i>Gammaproteobacteria</i>	<i>Enterobacterales</i>	<i>Erwinia</i>	12.6	5.3	13.4	2.1	12.6	0.0
<i>Pseudomonadota</i>	<i>Acidiphilium</i>	<i>Rhodospirillales</i>	<i>Acidiphilium</i>	0.5	9.4	1.2	12.2	0.9	0.1
<i>Pseudomonadota</i>	<i>Gammaproteobacteria</i>	<i>Enterobacterales</i>	<i>Citrobacter</i>	0.5	2.5	0.9	2.2	1.1	0.1
<i>Proteobacteria</i>	<i>Gammaproteobacteria</i>	<i>Enterobacterales</i>	<i>Enterobacter</i>	2.3	5.2	1.2	2.2	0.9	8.2
<i>Proteobacteria</i>	<i>Alphaproteobacteria</i>	<i>Rhodospirillales</i>	<i>Gluconobacter</i>	0.3	12.4	0.3	5.2	1.1	15.2
<i>Pseudomonadota</i>	<i>Alphaproteobacteria</i>	<i>Rhodospirillales</i>	<i>Acetobacter</i>	0.4	13.5	1.3	18.6	0.2	5.7
<i>Pseudomonadota</i>	<i>Gamma Proteobacteria</i>	<i>Enterobacterales</i>	<i>Serratia</i>	0.2	13.4	0.1	12.4	1.1	22.5

If apple pomace is to be reused for feedstuffs it should be during the first months of storage, as fiber and protein decreased over time and the recommended values of these parameters change depending on the diet being made for ruminant or non-ruminant species.

Likewise, if apple pomace is used as material for composting, it should be before protein values start to decrease as it was showed previously. As proteins are an important source of nitrogen, necessary for supporting the growth and metabolism of microorganisms in composting processes.

In the case of pellet production, it was mentioned that independently of storage conditions the calorific value of the samples remains stable over time. This makes the use of this residue as pellet material a viable option at first sight, especially in longer storage times under-roof conditions, where the moisture content is adequate for pellet production. For that reason, it is recommended to reuse apple pomace just before 6 months of storage when moisture content is low, and the odorous compounds have not reached odor thresholds. Considering all previous scenarios and the results obtained, it is recommended that apple pomace always gets stored in Under-roof conditions, as it is the one that generates the lesser impact on VOCs production and its

4. Conclusions

Results of this study shed light on the dynamic evolution of apple pomace under different storage conditions, highlighting the interplay of physico-chemical and biological changes. Outside temperature, relative humidity, and precipitation were found to be key factors influencing these transformations. The analysis of VOCs and microorganisms demonstrated a progressive decomposition of the waste, driven by chemical and biological mechanisms. The increased ecological diversity of bacteria and fungi further emphasized the organic breakdown.

The study's significance lies in addressing critical knowledge gaps regarding VOC emissions during apple waste storage and degradation. The correlation between storage conditions and odorant VOC emissions, microbial diversity changes, and physico-chemical alterations was thoroughly examined over an extended 8-month period. Results provide valuable insights for potential reuses of apple pomace, such as composting material, alternative feedstuffs, and bioproducts as long as pomace is kept under-roof and for periods shorter than six months. This is due to ester, aldehydes and hydrocarbon's derivatives exceeding odor threshold values beyond this period, posing environmental and health risks to local communities.

By comprehensively analyzing the temporal evolution of apple pomace under various storage conditions, this study contributes significantly to our understanding of decomposition mechanisms and implications. It offers a foundation for informed waste management decisions, aiding in sustainable practices and reducing environmental impact.

Author contribution statement

Diógenes Hernández Espinoza: Conceived and designed the experiments; Performed the experiments; Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data; Wrote the paper.

Carlos Zambra: Performed the experiments; Contributed reagents, materials, analysis tools or data.

David Gabriel: Performed the experiments; Analyzed and interpreted the data; Wrote the paper.

Cesar Astudillo Hernandez: Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data.

Joquín Díaz: Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data; Wrote the paper.

Data availability statement

The authors are unable or have chosen not to specify which data has been used.

Additional information

Supplementary content related to this article has been published online at [URL].

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

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