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Sustainable Use of Cultivated and Wild Capers in Morocco: A Comparative Study of Physicochemical, Microbiological, and Functional Properties During the Natural Fermentation Process

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Abstract

Traditional fermentation is considered a sustainable bioprocess for the transformation and preservation of local agri-food resources. In this context, the present study investigates the influence of Moroccan caperberries (*Capparis spinosa* L. complex) sources (wild and cultivated) on physicochemical, microbiological, and functional properties during a 60-day natural fermentation process, with a focus on sustainable food valorization, local resource utilization, and eco-friendly practices. The results revealed that the fermented Moroccan caperberries displayed favorable physicochemical and microbiological evolutions, characterized, respectively, by a progressive acidification and sugar consumption and a rapid inhibition of Enterobacteria and persistence of an important level of LAB count at the end of the fermentation. Moreover, the functional properties analyses showed that the values of vitamin C content, total polyphenols content, and antioxidant activities range between 0.09–1.25 mg/100 g, 5.69–243.99 mgGAE/L, and 21.33–71.06% for DPPH and 42.31–875.26 µgAAE/mL for FRAP, respectively. However, the results demonstrated that the provenance significantly influences the fermentation profile, providing a benefit for wild caperberries due to their elevated levels of vitamin C, polyphenol content, and antioxidant activities. The fermentation period of 15 to 23 days can be recommended for optimal functional quality, while longer fermentation (up to 60 days) could be preferable when extended shelf life and commercial stability are the primary objectives. From a sustainability perspective, these findings suggest a complementary strategy: cultivated *C. spinosa* complex should be further promoted to ensure a consistent raw material supply and food safety, while wild provenances, owing to their exceptional functional potential, represent valuable genetic resources that should be conserved and preferentially integrated into cultivation programs.

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Keywords: Moroccan caperberries; *Capparis spinosa* L. complex; natural fermentation; functional properties; sustainable food processing

1. Introduction

In the Mediterranean basin, fermentation represents a longstanding traditional practice for producing a wide range of fermented foods [1]. As an ancestral method of food preservation, it has gained renewed scientific and industrial interest [2]. This process enhances the preservation of various seasonal foods, including olives, prickly pears, grapes, and capers [3]. In Morocco, traditional fermented products are mainly obtained through spontaneous lactic fermentation [4], a widely applied approach that improves the preservation, safety, and nutritional value of fruits such as capers.

In Morocco, *Capparis spinosa* L. is grown mainly in the provinces of Taza, Fez, Marrakech, Meknes, and Safi [5], producing about 21,560 t/year of caperberries. The Moroccan caperberries were reported as having the best aesthetic appearance and morphological characteristics for industrialization [6] and are considered a rich source of phenolic compounds and antioxidants [7,8]. Recently, in an ethnobotanical study conducted in Taza Province, *C. spinosa* L. was identified as a spontaneous medicinal species [7,9], and its fruits are prepared based on the natural fermentation process [10]. This traditional knowledge needs to be transmitted and studied scientifically in the laboratory for the proper development of this sector. Moreover, previous studies reported *C. spinosa* L. as one of the most notable medicinal plants for the prevention and treatment of various diseases [11,12] and as an edible plant with health-promoting benefits due to its potential bioactive compounds [13]. The most consumed parts are capers (flower buds) and caperberries (fruits) [14,15]. The latter are rarely consumed fresh and require a transformation stage to reduce their glucocapparin, which is responsible for bitterness [16–18]. It consists of immersing the fresh fruits in brine [19,20], where they are subjected to natural lactic fermentation [21], occurring spontaneously and mainly by autochthonous LAB present in the raw material [1,22]. At the biochemical level, glucocapparin is enzymatically hydrolyzed by endogenous myrosinase in fruits into an unstable aglycone that is subsequently converted into various degradation products, mainly methyl isothiocyanate, a less persistent compound in the aqueous medium, which contributes to the reduction of perceived bitterness.

However, the caperberry sector represents an important component of the Moroccan economy. Its processing, both at artisanal (cooperatives and small producers) and industrial levels, primarily relies on brine fermentation. This process generally involves NaCl concentrations between 10 and 20%. Nevertheless, post-harvest practices often remain empirical, which can lead to qualitative and quantitative losses due to uncontrolled fermentation and microbial contamination. In this context, a better understanding of the microbiological and biochemical changes during fermentation, especially in the brine, could be essential to improve the quality of the final product and reduce post-harvest losses. However, despite the economic importance of Moroccan caperberries, current knowledge remains limited regarding how the origin of the raw material (wild vs. cultivated) influences these transformations and, consequently, the functional and microbiological quality of the final product. Addressing this gap is crucial for developing more standardized and efficient fermentation practices. This approach directly contributes to strengthening the added value of Moroccan capers, supporting the incomes of farmers and cooperatives, and promoting more sustainable and standardized processing practices.

From a sustainability perspective, natural lactic fermentation represents a low-input, environmentally friendly food processing strategy that supports the valorization of local

plant resources, reduces post-harvest losses, and preserves agrobiodiversity. The use of wild and cultivated sources of *C. spinosa* L. aligns with sustainable food systems by promoting the use of indigenous biodiversity and traditional knowledge while minimizing reliance on chemical additives. Although several previous works have studied the fermentation of caperberries [1,17,18,20–22], the novelty of our study constitutes the first comparison between the natural fermentation of wild and cultivated Moroccan caperberries through a simultaneous comparative study of physicochemical, microbiological, and functional properties. All the more so, the present work aimed to develop a novel fermented food product based on lactic-fermented caperberries with enhanced functional properties and potential health benefits for consumers. Specifically, this work evaluated the influence of caperberry sources (wild and cultivated) on the physicochemical characteristics, microbiological profile, and functional properties of fermented caperberries during the natural fermentation process.

2. Materials and Methods

2.1. Caperberry Fruit Origin

Fresh caperberries of *C. spinosa* complex, originating from two provenances: the wild from Taza Province and the cultivated one from Moulay Yacoub Province in North-Central Morocco [6], were hand-harvested in the early morning in June 2023 and were transported in cold bags to the laboratory. Then, they were sorted manually to obtain uniform colors and degrees of maturity. Based on previous work [7], the smallest size (<8 mm) was used in this study due to its aptness for processing and industrial relevance, as well as its richness in phenolic compounds and antioxidants.

2.2. Caperberry Fermentation Process

The caperberry fruits (wild and cultivated) were subjected to a washing stage, which corresponds to their immersion in water for 24 h (changing the water every 12 h), to reduce bitterness for successful lactic fermentation (reducing the effect of polyphenols on LAB). Then, under sterile working conditions, each lot of caperberries was introduced into sterilized flasks of 1 L, respectively, with a ratio of 300 g in 300 mL of sterilized distilled water initially brined at 10% NaCl (*m*/*v*). All caperberry fermentation processes were conducted in triplicate and incubated at room temperature for two months. During this fermentation period, the brine samples were tested aseptically and regularly on days 1, 8, 15, 23, 30, 38, 45, 53, and 60 for physicochemical and microbiological analysis and functional properties. All analyses were performed on identical biological replicates under the same experimental conditions.

2.3. Physicochemical Analysis

The physicochemical analysis (pH, free acidity, chlorides, and total reducing sugars) of caperberries was evaluated during the spontaneous fermentation process, following the methods of [23]. The pH of brine samples was measured using a Crison pH meter type pH 2000 (Crison Instruments, Barcelona, Spain) after calibration at pH 4 and 7. The free acidity was determined by titrating brine samples using NaOH (0.1 M) and phenolphthalein as indicators, and the results were expressed as a percent of lactic acid, according to the formula: $X = N\ (NaOH) \times V_{eq}\ (NaOH) \times M_{ac}/V_0$. {Where; X: Acidity in g/L; N: 0.1 mol/L of NaOH; V_{eq}: Volume of NaOH added; M_{ac}: Molar mass of lactic acid (90.08 g/mol); and V_0: Volume of sample brine}. The chlorides in brine samples were measured by titration with AgNO3 (0.1 M) in the presence of potassium chromate (0.5%, *w*/*v*) as an indicator until the appearance of the persistent brick red color, and the contents were expressed as a percentage of NaCl. The total reducing sugar contents were determined

using the 3,5-dinitrosalicylic acid (DNS) method. It consists of the reduction of DNS by reducing sugars present in the sample brine under alkaline conditions and heating in a boiling water bath (≈100 °C) for 10 min to form 3-amino-5-nitrosalicylic acid, which produces an orange-colored complex. After cooling to room temperature, the absorbance was measured at 540 nm. Quantification was performed using a calibration curve prepared with standard glucose solutions, and results were expressed as mM glucose equivalents. All physicochemical analyses were conducted in triplicate.

2.4. Microbiological Analysis

The microbiological analysis of brine samples included enumeration of total aerobic mesophilic flora (TAMF), Enterobacteria, lactic acid bacteria (LAB), and yeasts and molds (Y&M), as described by [23]. The brine samples were serially diluted in sterile saline solution. Microorganisms were enumerated using the plate count method. Each dilution was plated on specific culture media. The TAMF and LAB were counted, respectively, on Plate Count Agar (PCA, Biokar, Allonne, France) and on Man Rogosa and Sharpe Agar (MRS, Biokar), containing cycloheximide (0.01%), after incubation at 30 °C for 48 h. Enterobacteria were enumerated on Deoxycholate Lactose (DCL) Agar (Biokar) after incubation at 37 °C for 48 h. Y&M were enumerated on Potato Dextrose Agar (PDA, Biokar) after incubation at 25 °C for 72 h. All microbiological analyses were performed in triplicate.

2.5. Vitamin C Content

The vitamin C content in brine samples was determined using the method of [24]. The sample brines were filtered through muslin cloth to remove solid residues, then diluted in a volumetric flask to a final volume of 25 mL using 4% oxalic acid. Ascorbic acid content was determined using the 2,6-dichlorophenol indophenol (DCPIP) titration method as described by Rao and Deshpande (2006) [25]. For the assay, 5 mL of the ascorbic acid working solution (500 µg/5 mL) was mixed with 10 mL of 4% oxalic acid in a 100 mL conical flask and titrated with the dye solution until a stable pale pink color appeared (volume V1). Subsequently, 5 mL of the filtered sample brine was titrated under the same conditions to measure the volume of dye consumed (V2). The ascorbic acid content (mg/100 g) of the solutions was calculated using the following formula: Vit C = $(500 \times V_2 \times 25 \times 100) \div (V_1 \times 5 \times 5)$. {Where; 500: µg of standard ascorbic acid taken for titration; V_1: Volume of dye consumed by 500 µg of standard ascorbic acid; V_2: Volume of dye consumed by 5 mL of the sample test; 25: Total volume of the extract; 100: Ascorbic acid content/100 g of the sample; 5: Weight of the sample taken for extraction; 5: Volume of the test sample taken for titration}. The test was realized in triplicate.

2.6. Total Polyphenol Content (TPC)

The total polyphenol content was determined in brine samples as [23] using Folin–Ciocalteu reagent (Sigma-Aldrich, St. Louis, MO, USA) and 20% (*w*/*v*) sodium carbonate solution and incubated at room temperature for 20 min in dark conditions. The mixture was measured at 760 nm, and the quantification of TPC was expressed as gallic acid equivalent (mgGAE/L) using a gallic acid standard curve. All analyses were measured in triplicate.

2.7. DPPH (2,2-Diphenyl-2-picrylhydrazyl) Radical Scavenging Assay

The antioxidant potential of brine samples was evaluated by DPPH (2,2-diphenyl-2-picrylhydrazyl) using the method of [26], with some modifications. 1 mL of each brine sample or distilled water (control) was mixed with 0.5 mL of DPPH solution (0.1 mM) prepared in methanol and incubated for 30 min at room temperature under dark conditions, then analyzed at 517 nm using a spectrophotometer (Specuvisi UV/VIS, No RE1701008,

Analytik Jena, Jena, Germany). The DPPH scavenging activity (%) was calculated using the following equation:

$$\text{DPPH scavenging activity (\%)} = 1 - (\text{Absorbance sample}_{(\text{at 517 nm})} / \text{Absorbance control}_{(\text{at 517 nm})}) \times 100.$$

2.8. FRAP (Ferric Reducing Antioxidant Power) Assay

The antioxidant activity of brine samples was conducted using a FRAP method [27] with some modifications. This colorimetric assay measures the reduction of ferric ions (Fe^{3+}) to ferrous ions (Fe^{2+}), producing a color change quantified at 700 nm. For each brine extract solution (as prepared above), 250 µL was mixed with 1.25 mL phosphate buffer (0.2 M, pH 6.6) and 1.25 mL potassium ferricyanide (1%, *w*/*v*). After 30 min of incubation at 50 °C, 1.25 mL of trichloroacetic acid (10%, *m*/*v*) was added to stabilize the complexes. The mixture was centrifuged (3000 rpm, 10 min), and 1.25 mL of the supernatant was combined with 1.25 mL of distilled water and 0.25 mL $FeCl_3$ (0.1%, *m*/*v*). Absorbance was measured using a Specuvisi UV/VIS spectrophotometer (No. RE1701008) at 700 nm. Results were calculated from a calibration curve using ascorbic acid as a standard and expressed as µg of ascorbic acid equivalents per milliliter of solution (µgAAE/mL). Analyses were performed in triplicate to ensure accuracy.

2.9. Statistical Analysis

All experiments were conducted in triplicate. Statistical analyses included ANOVA, LSD test ($p < 0.05$), Pearson correlation, and PCA. The statistical analysis performed using the STATGRAPHICS Centurion XVII package (Version 8, Stat Point Technologies, Inc., Warrenton, VI, USA) was used for all the calculations. The General Linear Models were carried out over time for the process and the provenance of caperberries. Analysis of variance (ANOVA) was performed, and means were compared. The least significant difference (LSD) values were calculated at the 5% probability level to determine which levels of the factors influence the dependent variables analyzed. A correlation (Pearson) matrix was performed based on the mean values of the parameters studied using Minitab 18 statistical software. Principal component analysis (PCA) was adopted in this current study as a multivariate statistical tool to represent the variability of parameters studied using XLSTAT software (XLSTAT Version 2016.02.28 451).

3. Results

3.1. Physicochemical Analysis

The results of physicochemical analysis (pH, free acidity, chloride content, and total reducing sugars) obtained during the natural fermentation process of wild (Wi) and cultivated (Cu) caperberries are presented in Figures 1 and 2 and Table 1. The pH showed a high decrease from 5.8 and 5.86 to 4.3 and 4.37 during the first two weeks of the fermentation process, respectively, for cultivated and wild caperberries (Figure 1). Then, the pH wavers progressively to stabilize at the end of the fermentation process at about 4.34 in cultivated caperberries and 4.6 in wild ones (Figure 1). Moreover, free acidity increased to 0.27% for cultivated caperberries and to 0.19% for wild ones during the first week of the fermentation, respectively (Figure 1), and a high mean value of free acidity (about 0.5%) was obtained at the end of the fermentation process. Therefore, this simultaneous decrease in pH and increase in free acidity can be attributed to the metabolic activity of LAB, which ferment available carbohydrates into organic acids, mainly lactic acid, leading to proton release and progressive acidification of the medium. In terms of provenance, a significant decrease in pH (4.5) and an increase in free acidity (0.397%) were observed in cultivated caperberries, compared to those (pH 4.71 and free acidity 0.357%) found in wild ones. The results of

ANOVA analysis (Table 2) showed that the effect of provenance affected the variabilities of free acidity by 10% (0.02 ***) and of pH by 32% (0.62 ***).

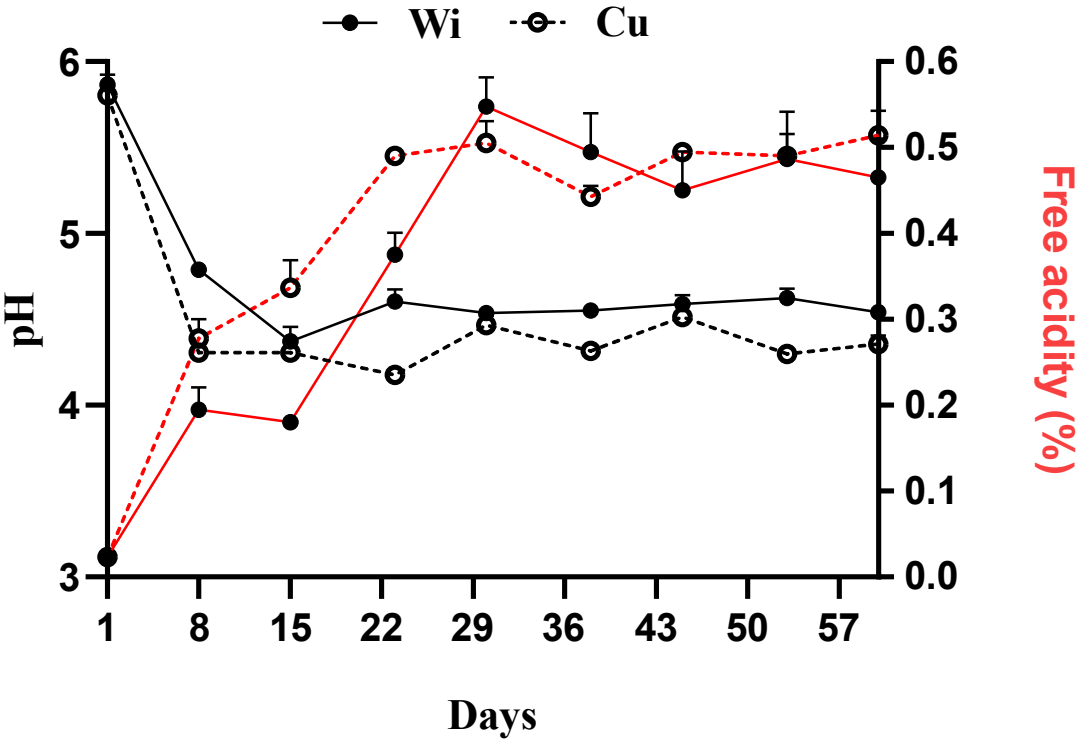

Figure 1. Evolution of pH (black color) and free acidity (%) (red color) during the natural fermentation process of wild (Wi ⊸) and cultivated (Cu: ⊸) caperberries.

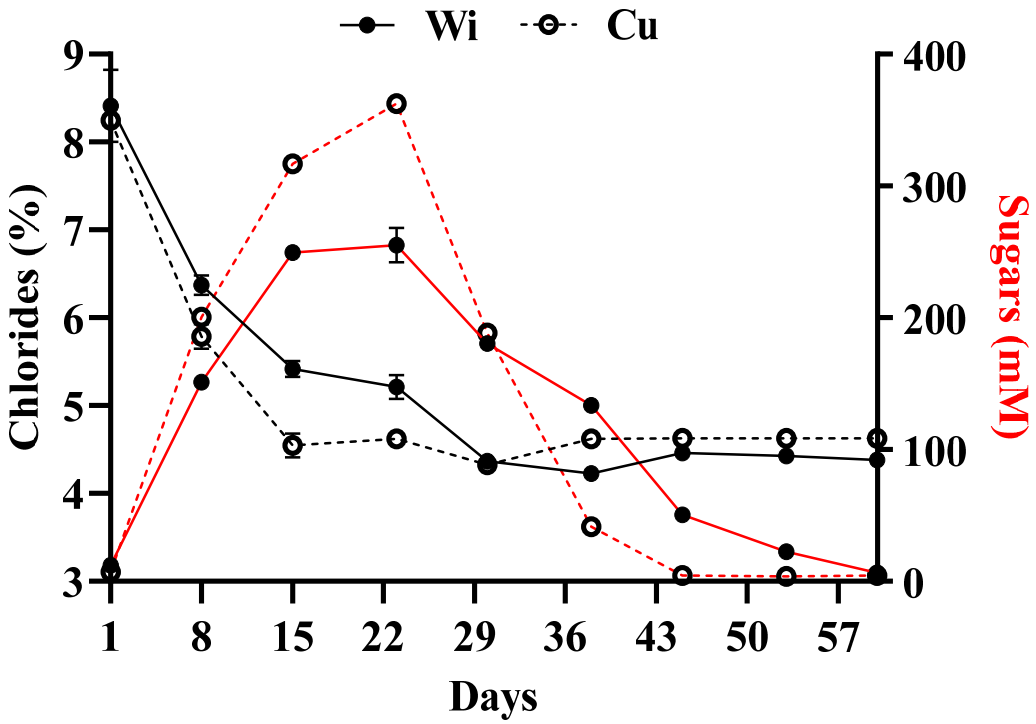

Figure 2. Changes in chlorides (%) (black color) and total sugar content (mM) (red color) during the natural fermentation process of wild (Wi ⊸) and cultivated (Cu: ⊸) caperberries.

Table 1. Mean values of all parameter changes (pH, free acidity (acid) in %, chlorides (Chl) in %, polyphenol content (phenols) in mgGAE/L, total reducing sugars content in mM, vitamin C (Vit C) in mg/100 g, FRAP assay in µg AAE/mL, DPPH AI in %, populations of LAB, Y&M, Entero and TAMF (expressed in Log cfu/mL)), monitored during the spontaneous natural fermentation of caperberries under factors of provenance (P) (wild (Wi) and cultivated (Cu)), and of time of process (T).

Factors		pH	Acid	Chl	Sugars	Vit C	Phenols	DPPH	FRAP	LAB	Y&M	Entero	TAMF
			(%)	(%)	(mM)	(mg/100 g)	(mgGAE/L)	(%)	(µgAAE/mL)		(Log cfu/mL)		
P	Wi	4.72 ^{a}	0.36 ^{b}	5.25 ^{a}	117.96 ^{b}	0.63 ^{a}	181.08 ^{a}	46.24 ^{a}	473.13 ^{a}	4.64 ^{b}	3.16 ^{b}	0.05 ^{a}	5.60 ^{b}
	Cu	4.50 ^{b}	0.40 ^{a}	5.12 ^{b}	125.54 ^{a}	0.56 ^{b}	166.87 ^{b}	46.04 ^{b}	469.15 ^{b}	5.06 ^{a}	3.82 ^{a}	0.07 ^{a}	5.95 ^{a}
T	1	5.83 ^{a}	0.02 ^{e}	8.33 ^{a}	9.53 ^{g}	0.09 ^{h}	5.69 ^{h}	21.33 ^{h}	42.31 ^{i}	4.33 ^{f}	3.71 ^{d}	0.55 ^{a}	5.03 ^{f}
	8	4.54 ^{b}	0.24 ^{d}	6.08 ^{b}	175.92 ^{d}	1.25 ^{a}	116.04 ^{g}	44.30 ^{e}	171.15 ^{h}	5.24 ^{d}	4.44 ^{c}	-	6.40 ^{d}
	15	4.33 ^{f}	0.26 ^{c}	4.98 ^{c}	283.18 ^{b}	0.91 ^{b}	243.99 ^{a}	71.06 ^{a}	875.26 ^{a}	5.93 ^{b}	5.40 ^{a}	-	7.30 ^{b}
	23	4.38 ^{e}	0.43 ^{c}	4.92 ^{c}	308.93 ^{a}	0.70 ^{c}	191.54 ^{e}	41.12 ^{g}	473.85 ^{g}	6.45 ^{a}	5.20 ^{b}	-	7.95 ^{a}
	30	4.5 ^{bc}	0.53 ^{a}	4.35 ^{e}	184.49 ^{c}	0.60 ^{d}	177.59 ^{f}	41.45 ^{f}	506.03 ^{f}	5.46 ^{c}	4.33 ^{c}	-	6.74 ^{c}
	38	4.43 ^{de}	0.47 ^{b}	4.42 ^{de}	87.47 ^{e}	0.61 ^{d}	182.73 ^{f}	52.41 ^{b}	572.44 ^{b}	4.99 ^{e}	3.20 ^{e}	-	5.78 ^{e}
	45	4.55 ^{b}	0.47 ^{b}	4.55 ^{d}	27.64 ^{f}	0.48 ^{e}	224.88 ^{b}	48.58 ^{c}	552.44 ^{c}	4.30 ^{f}	2.30 ^{f}	-	4.92 ^{g}
	53	4.46 ^{cd}	0.49 ^{b}	4.53 ^{d}	13.16 ^{g}	0.41 ^{f}	208.11 ^{d}	47.66 ^{d}	518.72 ^{e}	3.58 ^{g}	1.47 ^{g}	-	4.03 ^{h}
	60	4.44 ^{d}	0.49 ^{b}	4.51 ^{d}	5.47 ^{h}	0.31 ^{g}	215.35 ^{c}	47.38 ^{d}	528.08 ^{d}	3.34 ^{h}	1.37 ^{g}	-	3.81 ^{i}

Mean values in each column followed by the same letter (^{a}, ^{b}, ^{c}, ^{d}, ^{e}, ^{f}, ^{g}, ^{h}, ^{i}) are not significantly different according to the LSD test at $p < 0.05$. -: no growth.

Table 2. Analysis of variance (ANOVA) for all parameters changes (pH, Free acidity (Acid) in %, chlorides (Chl) in %, Polyphenols content (Phenols) in mgGAE/L, total reducing sugars content in mM, Vit C in mg/100 g, FRAP assay in µg AAE/mL, DPPH AI in %, Populations of LAB, Y&M, Entero and TAMF (Log cfu/mL)), monitored during the spontaneous natural fermentation of caperberries under factors of Provenance (P) (wild (Wi) and Cultivated (Cu)), and of Time of process (T).

Factors	Df	pH	Free Acidity	Chlorides	Total Reducing Sugars	Polyhenols	Vit C	DPPH AI	FRAP	LAB	Y&M	Entero	TAMF
			(%)	(%)	(mM)	(mgGAE/L)	mg/100 g	(%)	µg AAE/mL		Log cfu/mL		
T	8	1.29 ***	0.17 ***	10.03 ***	86,930 ***	1,099,560 ***	0.69 ***	1002 ***	345,599 ***	6.54 ***	13.74 ***	0.20 ***	12.39 ***
P	1	0.62 ***	0.02 ***	0.26 ***	776.4 ***	94,140 ***	0.07 ***	0.54 *	214.77 ***	2.42 ***	5.9 ***	0.01	1.61 ***
T × P	8	0.04 ***	0.01 ***	0.30 ***	5426 ***	146,687 ***	0.01 ***	4.56 ***	170.47 ***	0.99 ***	0.70 ***	0.01	0.68 ***
Rep	2	0.001	0.001	0.01	18.04	455.1	0.003	0.55 **	1.06	0.01	0.02	0.02	0.005
Res	34	0.001	0.001	0.015	10.83	1171.39	0.001	0.08	5.02	0.01	0.010	0.01	0.002

Df: Degrees of freedom; Rep: Repetition; Res: Residual; * Significant at 0.05 probability level, ** Significant at 0.01 probability level; *** Significant at 0.001 probability level.

In parallel, the results of chloride changes showed a significant decrease in mean values from 8.32% to 4.91% during the 15th day of the process (Table 1) and a continuous decrease to stabilize at the end of the process (4.5%) (Figure 2). Furthermore, the brine of wild caperberries showed significant differences ($p < 0.05$) in the mean value of chloride content (5.25%) compared to that (5.12%) in the brine of cultivated ones (Table 1). The results of total reducing sugars reported in Figure 2 showed an important increase during the 23rd day of the fermentation to achieve a maximal mean value (308 mM) (Table 1). This initial rise can be attributed to the diffusion and release of soluble sugars from caperberries into the brine, facilitated by cell wall degradation and osmotic processes. However, the progressive and continuous decrease of sugar content at the end of the fermentation process to 13 mM as the mean value could mainly be due to microbial consumption, particularly by the LAB population and other fermentative microorganisms, which utilize these sugars as carbon and energy sources for growth and organic acid production.

Furthermore, the results of the combined ANOVA (Table 2) showed the predominant effect of the time of process by 93% (86,930 ***) on the variance of sugars, and the interaction effect between time of process and provenance presented 6% (5426 ***) on the variability of sugar. While even the effect of provenance was the lowest and did not exceed 1% (776.4 ***) on the variance of sugars, significant differences in their content were obtained due to the effect of provenance (Table 1), and the highest mean value of total reducing sugars (125.544 mM) was found in the brine of cultivated caperberries compared to that (117.961 mM) found in wild ones.

3.2. Microbiological Analysis

The results of the microbiological analysis of brine samples are presented in Figure 3 and Table 1. The TAMF count was increased during the fermentation period and reached a maximum of 7.95 Log cfu/mL on the 23rd day, and then decreased progressively to a mean value of 3.81 Log cfu/mL at the end of the fermentation process (Table 1). Moreover, a significant difference ($p < 0.05$) was found (Table 1) with the highest mean value of 5.95 Log cfu/mL in cultivated caperberries compared to 5.6 Log cfu/mL in wild ones.

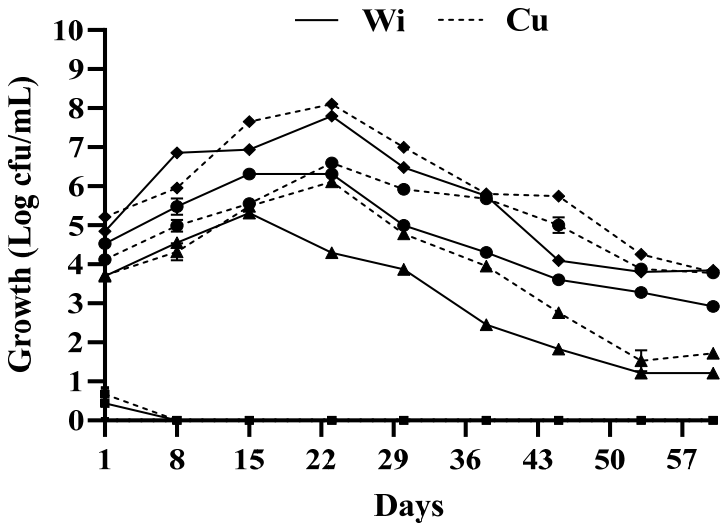

Figure 3. Evolution of TAMF (♦), LAB (●), Y&M (▲), and Enterobacteriaceae (■) populations (expressed in Log cfu/mL) during the natural fermentation process of wild (— Wi) and cultivated (---- Cu) caperberries.

The LAB count was increased until a maximum growth mean value of 6.45 Log cfu/mL on the 23rd day of the process, followed by a continuous decrease to a mean value of 3.34 Log cfu/mL at the end of the fermentation process (Table 1). Furthermore, a signifi-

cant difference ($p < 0.05$) in the mean value of LAB was observed in cultivated caperberries (5.06 Log cfu/mL) compared to 4.64 Log cfu/mL obtained in wild caperberries.

The Y&M count showed an increase to the mean value of 5.4 Log cfu/mL during the first 15 days of the process; then, they underwent a significant continuous decrease ($p < 0.05$) to around 1.37 Log cfu/mL at the end of the fermentation (Figure 3, Table 1). Moreover, the significant differences ($p < 0.05$) were observed between the two provenances, and the highest mean value of 3.82 Log cfu/mL was obtained in cultivated caperberries compared to 3.16 Log cfu/mL found in wild ones (Table 1).

On the other hand, the Enterobacteria presented the initial counts of around 1 Log cfu/mL and disappeared completely on the eight day of the fermentation process in two provenances. This elimination can be attributed to the combined effects of acidification and competitive exclusion by LAB, which dominate the fermentation ecosystem. The rapid elimination of Enterobacteria is a key indicator of fermentation safety, as it reflects the inhibition of potentially undesirable or pathogenic microorganisms. However, no significant difference ($p < 0.05$) was observed between cultivated and wild caperberries (Table 1). The combined ANOVA (Table 2) revealed the dominant effect of time of the process to be about 84.4% (12.39 ***), 65.6% (6.54 ***), 67% (13.74 ***), and 82% (0.2 ***) on the total observed variance of the TAMF, LAB, Y&M, and Enterobacteria populations, respectively. However, the effects of provenance and the interaction between two factors on the variability of TAMF were 11% (1.61 ***) and 4.6% (0.68 ***), respectively, 24% (2.42 ***) and 10% (0.99 ***), respectively, for LAB, 29% (5.9 ***) and 3.4% (0.7 ***) for Y&M.

3.3. Vitamin C Content

The results of the vitamin C content during the fermentation process of caperberries are presented in Figure 4 and Table 1. The vitamin C content in caperberry brine increased in the first week of fermentation and reached the maximal mean value on the eighth day of the process (1.251 mg/100 g), followed by a progressive decrease to 0.3 mg/100 g at the end of the process. A significant difference ($p < 0.05$) in vitamin C was observed when comparing wild caperberries (0.633 mg/100 g) to cultivated ones (0.56 mg/100 g) (Table 1). Therefore, ANOVA analysis showed the predominant effect of the process time at 89% (0.69 ***) and a slight effect of provenance at 10% (0.07 ***) on the total variance of vitamin C (Table 2).

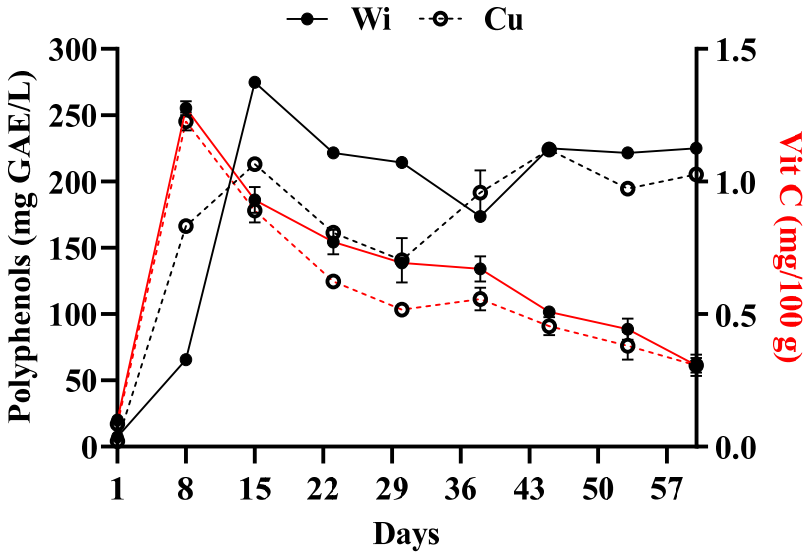

Figure 4. Evolution of total polyphenol (mgGAE/L) (black color) and vitamin C (mg/100 g) contents (red color) during the natural fermentation process of wild (Wi: —●—) and cultivated (Cu: ···○···) caperberries.

3.4. Total Polyphenol Content

The results of the total polyphenol content (TPC) of the brine during fermentation are shown in Figure 4 and Table 1. They increased rapidly to achieve 243.99 mgGAE/L as the mean value during the 15th day of the fermentation, followed by a progressive decrease to about 214.35 mgGAE/L at the end of the fermentation process. A significant difference in TPC (Table 1) was obtained between wild caperberries (181.08 mgGAE/L) and cultivated ones (166.87 mgGAE/L). The results of the combined ANOVA (Table 2) showed the predominant effect of time of process with 82% (1,099,560 ***) on the total variance of TPC. The effect of the provenance was lower and did not exceed 7% (94,140 ***). The effect of the interaction between the time of the process and provenance showed an important effect of 11% (146,687 ***) on the variance of this parameter.

3.5. Antioxidant Activity

The results of DPPH and FRAP assays during the fermentation process of caperberries are presented in Figure 5 and Table 1. The scavenging effect increased drastically during the first 15 days of the fermentation to achieve a maximal mean value (71%, 875 µgAAE/mL) of DPPH and FRAP assays, respectively, followed by their progressive decrease to stabilize at around (47%, 520 µgAAE/mL) at the end of the fermentation process (Figure 5). Furthermore, the results of the antioxidant activity of brine samples based on DPPH and FRAP assays showed significant differences ($p < 0.05$) during the time of the fermentation process (Table 1).

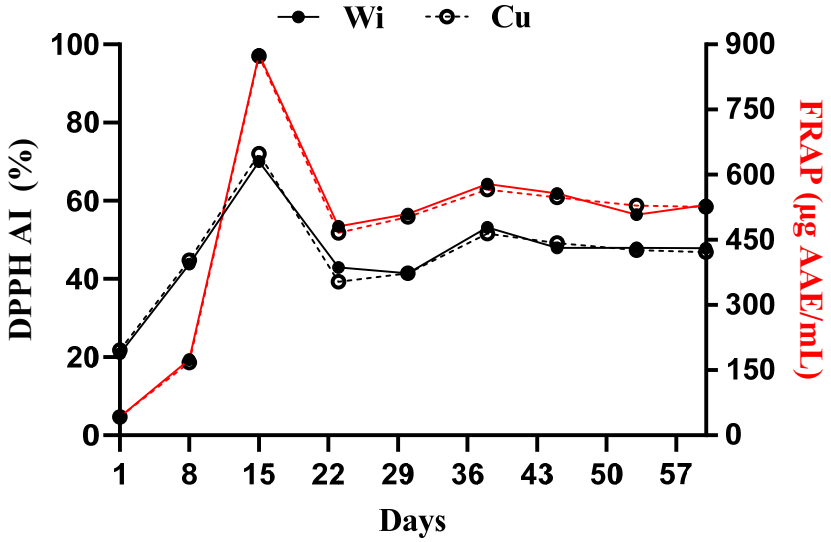

Figure 5. Evolution of antioxidant activity of caperberry brines (based on DPPH AI (%) (black color) and FRAP assay (µgAAE/mL) (red color)) during the natural fermentation process of wild (Wi: ⊸) and cultivated (Cu: ⊸) caperberries.

The combined ANOVA (Table 2) showed the predominant effect of the time of process, explaining 99% of the total variance of DPPH (1002 ***) and of the FRAP assay (345,599 ***). Even the effects of provenance on the variability of antioxidant activity were very low and did not exceed more than 0.06% (0.54 * for DPPH and 214.77 *** for FRAP) (Table 2); significant differences ($p < 0.05$) in mean values of DPPH and FRAP assays (46.24% and 473.13 µgAAE/mL) were obtained in wild caperberries compared to (46.04% and 469.15 µgAAE/mL) found in cultivated ones.

3.6. Correlation Matrix (Pearson)

Table 3 presents Pearson correlation coefficients among physicochemical, functional, and microbiological parameters during the fermentation process. A total of 42.42% of the

correlations are statistically significant, comprising 10.61% at $p < 0.05$, 10.61% at $p < 0.01$, and 21.21% at $p < 0.001$. The pH shows a strong negative correlation with acidity (r = –0.765 **), while it is positively correlated with Enterobacteriaceae (r = 0.978 ***). Acidity is significantly negatively correlated with chlorides (r = –0.939 ***) and Enterobacteriaceae (r = –0.790 ***) and positively correlated with phenolic content (r = 0.760 **).

Table 3. Pearson's correlation matrix among all parameter changes (pH, free acidity (acid) in %, chlorides (Chl) in %, polyphenol content (phenols) in mgGAE/L, total reducing sugar content in mM, vitamin C (Vit C) in mg/100 g, FRAP assay in µgAAE/mL, DPPH AI in %, populations of LAB, Y&M, Entero, and TAMF (Log cfu/mL), monitored during the spontaneous natural fermentation of caperberries under factors of provenance (P) (wild (Wi) and cultivated (Cu)) and of time of process (T).

Variables	Acid	Chl	Sugars	Vit C	Phenols	DPPH	FRAP	LAB	Y&M	Entero	TAMF
	%	%	mM	mg/100 g	mgGAE/L	%	µgAAE/mL		(Log cfu/mL)		
pH	−0.765 **	0.907 ***	−0.419	−0.540	−0.882 ***	−0.768 **	−0.735 *	−0.341	−0.018	0.978 ***	−0.206
Acid		−0.939 ***	−0.024	−0.008	0.760 **	0.361	0.557	−0.031	−0.390	−0.790 **	−0.159
Chl			−0.164	−0.204	−0.916 ***	−0.656 *	−0.776 **	−0.124	0.234	0.911 ***	0.015
Sugars				0.691 *	0.241	0.379	0.333	0.969 ***	0.886 ***	−0.349	0.938 ***
Vit C					0.240	0.506	0.176	0.624 *	0.576	−0.559	0.616 *
Phenols						0.823 **	0.907 ***	0.163	−0.169	−0.866 **	0.003
DPPH							0.889 ***	0.282	0.110	−0.720 *	0.185
FRAP								0.270	0.028	−0.670 *	0.147
LAB									0.912 ***	−0.272	0.981 ***
Y&M										0.057	0.964 ***
Entero											−0.141

* Significant at 0.05 probability level; ** Significant at 0.01 probability level; *** Significant at 0.001 probability level.

Chlorides (Chl) show strong negative correlations with polyphenols (r = –0.916 ***), while being strongly positively correlated with Enterobacteriaceae (r = 0.911 ***). Sugars showed a strong positive correlation with LAB (r = 0.969 ***), Y&M (r = 0.886 ***), and TAMF (r = 0.938 ***). Vitamin C shows positive correlations with LAB (r = 0.624 *) and TAMF (r = 0.616 *). Polyphenols are positively correlated with DPPH (r = 0.823 **) and FRAP (r = 0.907 ***), but they showed a significant negative correlation with Enterobacteriaceae (r = –0.866 **). Significant and positive correlations of TPC and DPPH (r = 0.823 **) and FRAP (r = 0.907 ***) and between DPPH AI% and FRAP (r = 0.889 ***) are obtained. Finally, LAB shows strong positive correlations with Y&M (r = 0.912 ***) and TAMF (r = 0.981 ***).

3.7. Principal Component Analysis (PCA)

The factorial analyses carried out by PCA based on the various physicochemical, functional, and microbiological parameters characterizing the natural fermentation process of wild and cultivated caperberries are illustrated in Figure 6. Figure 6A presents the correlations among variables. The first two principal components (PC1 and PC2), which together explain 86.71% of the total variance (PC1 = 52.54% and PC2 = 34.17%), provide a reliable and robust representation of the data structure.

PC1 is strongly influenced by pH, chloride content (Chl), and Enterobacteriaceae and, to a lesser extent, by polyphenols, DPPH, FRAP, and acidity. PC2, meanwhile, is shaped mainly by TAMF, Y&M, LAB, sugars, and vitamin C. Significant negative correlations are observed between polyphenols and free acidity versus chlorides and Enterobacteriaceae, respectively. Conversely, positive and significant correlations are found between acidity and phenols, as well as between FRAP and DPPH.

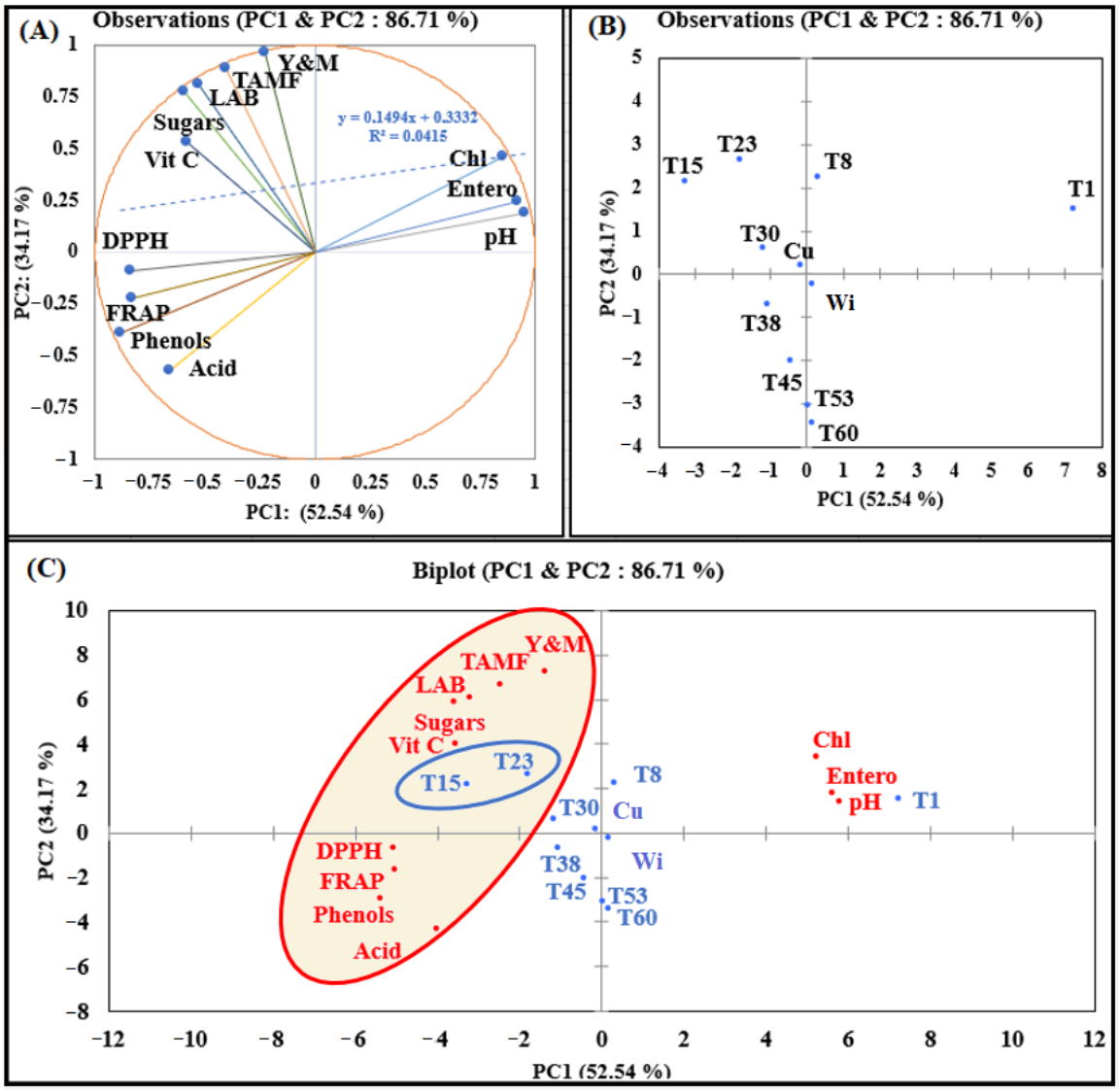

Figure 6. Principal component analysis (PCA). (**A**) Correlation circle showing relationships among biochemical, physicochemical, and microbiological parameters during caperberry fermentation. PC1 (52.54% of total variance) separates samples according to acidity and antioxidant activity (DPPH, FRAP, and phenols) versus pH and microbial safety indicators (Enterobacteriaceae and chlorides). PC2 (34.17% of variance) is mainly associated with microbial activity (LAB, TAMF, and Y&M) and nutrient availability (sugars). (**B**) Distribution of samples according to provenance (Wi: wild; Cu: cultivated) and fermentation time (T1–T60). (**C**) Biplot illustrating the relationship between variables and observations. Colored areas indicate favorable fermentation conditions.

Furthermore, sugars, LAB, Y&M, TAMF, and vitamin C are positively correlated and cluster together. In Figure 6B, which shows the distribution of observations (samples) across PC1 and PC2, the fermentation stages are clearly distinguished. The sample at T1 (day 1) is separated from all others. Intermediate stages, such as T15 and T23, are positioned near microbial and biochemical variables. In contrast, late stages (T53 and T60) are associated with low pH. Figure 6C (biplot) combines both variables and observations, reinforcing the interpretation of Figure 6A,B. A clear grouping is observed between T15 and T23, closely aligned with sugars, LAB, TAMF, and vitamin C. In contrast, T1, along with Chl, pH, and Enterobacteriaceae, forms a cluster on the opposite side. The shaded area highlights the region of optimal fermentation conditions.

4. Discussion

The present work highlights, for the first time, a comparison between the natural fermentation of wild and cultivated Moroccan caperberries through the evolution of physicochemical, microbiological, and functional properties. The fermentation process of wild and cultivated caperberries showed a change in physicochemical properties. The found values of pH decreased (4.34 and 4.6) and of free acidity increased (about 0.5%) obtained at the end of the fermentation process of cultivated and wild caperberries, respectively, are in accordance with those reported in previous studies [17,21,28] and remain more suitable than those (pH 5.19 and free acidity 0.18%) reported in caperberries brine in another study [29]. This simultaneous decrease in pH and increase in free acidity can be attributed to the accumulation of organic acids produced through sugar metabolism by the autochthonous LAB population. Indeed, the significant differences in pH (4.5) and in free acidity (0.397%) in cultivated caperberries, compared to those (pH 4.71 and free acidity 0.357%) in wild ones due to the effect of provenance, could be attributed to the significant differences in richness in chemical composition of raw material (mainly sugars and polyphenols) and in indigenous microbiota of LAB. The decrease in pH and increase in free acidity can be directly interpreted in relation to the measured evolution of LAB populations in our system. In particular, the more pronounced acidification observed in cultivated caperberries compared to wild ones is explicitly associated with their higher initial LAB counts and greater availability of fermentable substrates (higher sugar content and lower polyphenol levels). This combination likely promoted a faster transition to the exponential growth phase of LAB, resulting in increased organic acid production. In contrast, the relatively higher polyphenol content in wild caperberries (known for its antimicrobial effects) may have partially limited LAB activity, leading to slower acidification kinetics. These interpretations are now directly supported by the temporal evolution of microbial counts and compositional differences observed in our samples. These results highlight the influence of raw material origin on LAB-driven acidification dynamics and, consequently, on the safety and preservation of the final product.

The results of the chlorides content decrease (4.5%) could be explained by their infiltration from brine into caperberries via osmotic exchange, which is of great interest to accelerate the debittering process (glucocapparin release) as well as to facilitate the release of nutrients (namely sugars, polyphenols, and vitamins) highly desired for the growth of microbiota, mainly LAB, which are beneficial microorganisms responsible for the fermentation process of caperberries [30]. Furthermore, the significant difference ($p < 0.05$) in the mean value of chloride content due to the provenance could be attributed to the degree of diffusibility for salts and to the chemical composition richness between the two provenances, which could be attributed to the significant differences in the morphology of the two origins [6].

On the other hand, the significant increase in total reducing sugars during the 23rd day of the fermentation indicates that their accumulation in brines, based on the osmosis process, exceeds their degradation. This initial increase in total reducing sugars is directly correlated with the increase in the number of LABs and their metabolic activity. As fermentation progresses, the subsequent decrease in sugar concentration is explicitly correlated with intense metabolic activity of LAB (sugar utilization predominates over their diffusion), which uses these sugars as carbon and energy sources. During this period, fermentable sugars are converted into organic acids, mainly lactic acid, leading to acidification of the medium. This stage corresponds to the phase of exponential growth of LAB, where sugar consumption is highest. In the later stages of fermentation, sugar concentrations stabilize at low levels due to substrate depletion and a slowdown in microbial activity (coinciding with the stationary phase of LAB), due to the inhibitory effects of salts, organic acids, and

polyphenols accumulated during this phase. Thus, the temporal evolution of sugar content reflects the dynamic balance between mass transfer phenomena and microbial utilization and can be considered an indicator of the progression of fermentation and metabolic activity. Similar results demonstrated a reduction of 70.65% of sugar in brined caperberries during lactic fermentation [29]. The significant difference observed in total reducing sugars due to the effect of provenance is in agreement with that found among some wild and cultivated berry species [31]. It is important to note that the differences between wild and cultivated capers are now better understood in light of microbial load and substrate availability. The greater sugar consumption observed in cultivated samples is consistent with their higher level of LAB and greater availability of fermentable sugars, while the slower decrease in sugar in wild samples could be related to lower microbial activity and the potential inhibitory effect of higher polyphenol content.

The results of efficient use of sugars by indigenous microorganisms reflect an optimized bioprocess resulting in good accumulation of free acidity, which suggests that spontaneous fermentation may represent a promising approach to reduce reliance on added organic acids. From a sustainability perspective, this natural acidification process highlights the potential of spontaneous fermentation as an ecological preservation strategy that minimizes the need for chemical additives while ensuring food safety and extending shelf life.

The results of maximal TAMF count values (7.95 Log cfu/mL) observed on the 23rd day and of their progressive decrease (3.81 Log cfu/mL) at the end of the fermentation process are similar to those reported [17] in caper bud fermentation, ranging between 4.6 and 6.9 log cfu/mL at the beginning of fermentation and between 3.0 and 4.4 log cfu/mL at week 6 of the fermentation process. The initial increase in TAMF corresponds to the adaptation and rapid proliferation of this microbiota originating from the raw material and environment, supported by the availability of nutrients released into the brine. As fermentation progresses, the progressive acidification of the medium, driven by LAB, along with increasing salt concentration, creates a selective pressure that inhibits the growth of non-acid-tolerant and spoilage microorganisms. The subsequent decrease in TAMF counts can therefore be attributed to the combined effects of low pH, osmotic stress, and microbial competition, leading to the dominance of more adapted populations, particularly LAB. This shift reflects the transition from a heterogeneous microbial community to a more stable and selective ecosystem characteristic of the later stages of fermentation. Overall, these microbial dynamics highlight the self-regulating nature of spontaneous fermentation, in which intrinsic physicochemical factors and microbial interactions contribute to product stabilization and safety without the need for synthetic preservatives.

The presence of LAB during the first 23 days of the process at a high level (6.45 Log cfu/mL) allows an important technological interest to ensure the natural lactic fermentation process of caperberries, which produces changes in both the profile and bioactive compounds [2]. However, their decrease at the end of the process to a (3.34 Log cfu/mL) could be related to nutrient depletion (mainly sugars) and also can be attributed to the accumulation of antimicrobial compounds such as polyphenols and their derivatives (glucosinolates and isothiocyanates). In this study, the persistence of LAB at 60 days of the fermentation process, with a level of 3.34 Log cfu/mL, remains higher than those (2.81 cfu/mL and 3.06 Log cfu/mL) found at 33 days of the process [28] in untreated capers and inoculated capers with *Lactobacillus pentosus*, respectively, and were not more detected at the 45th day of the fermentation process. Consequently, the persistence of LAB in our study may be attributed to the reduction of the high amounts of polyphenols before the fermentation process, which is of great technological interest, allowing for the production of naturally fermented products with probiotic properties.

The Y&M counts found at the end of this process (1.37 Log cfu/mL) remain lower than that (3.21 Log cfu/mL) reported in untreated capers [28] and lower than that detected at the end of the fermentation process [1]. This reduction can be explained by several possible inhibitory factors that develop during fermentation, such as the progressive acidification of the brine due to lactic acid production by LAB, which leads to a decrease in pH, creating unfavorable conditions for the growth of many Y&M. The brine exerts osmotic stress, limiting the proliferation of salt-sensitive fungal species. The depletion of readily fermentable sugars reduces the availability of essential substrates required for yeast metabolism. Furthermore, LAB may contribute to this inhibition through the production of antimicrobial compounds such as organic acids, hydrogen peroxide, and, in some cases, bacteriocin-like substances, which can indirectly affect fungal populations. Competitive interactions for nutrients and ecological niches also play a key role in restricting Y&M growth. Altogether, these possible combined effects lead to a selective environment that suppresses spoilage-associated fungi, thereby improving the organoleptic quality and microbiological stability of the final product [32].

The presence of Enterobacteria at low levels and their total disappearance on the eighth day of the fermentation process in two provenances highlights a critical step in the microbiological stabilization of the system. In other works on spontaneous fermentation of caperberries, the disappearance of this microbiota was observed on the first day of fermentation due to the drastic drop in pH from 7.54 to 4.4 [21] and from 7.50 to 4.37 [1], achieved within the first day of fermentation. In our study, this rapid decline can be attributed first to the antibacterial effect of polyphenols, as was previously reported for *C. spinosa* complex parts against pathogenic bacteria [33], and also to the synergic antimicrobial effects of various parameters such as salt, low pH, high acidity, and polyphenols accumulations at the beginning of the fermentation process. Beyond these individual effects, the elimination of Enterobacteria also reflects strong microbial competition within the fermenting ecosystem. As LAB proliferate, they outcompete undesirable and potentially pathogenic microorganisms through rapid acid production and the creation of unfavorable environmental conditions. This competitive exclusion is a key mechanism driving the transition from a heterogeneous microbial community to a more selective and stable LAB-dominated system. Importantly, the complete disappearance of Enterobacteria during fermentation has direct implications for product safety, as it indicates the effective suppression of potential pathogens through natural fermentation processes. This self-regulating microbial dynamic could reinforce the role of spontaneous fermentation as a strategy to guarantee food safety without resorting to chemical preservatives.

The significant differences ($p < 0.05$) observed in TAMF counts, LAB counts, and Y&M counts due to the effect of provenance (the highest mean values of these populations were obtained in cultivated caperberries compared to those found in wild ones) could be explained by the richness and the significant differences in chemical composition (mainly polyphenols and sugars) of raw material provenances. In fact, cultivated caperberries exhibited a significantly higher initial load of LAB, along with a higher content of reducing sugars and a lower level of TPC compared to wild ones. This combination of factors likely favored a more active fermentation, characterized by enhanced microbial metabolism of sugars into organic acids, leading to a greater acidification of the medium. Conversely, the relatively higher TPC in wild caperberries may have contributed to a moderating effect on microbial activity (low level of LAB), given the known antimicrobial properties of certain phenolic compounds. Therefore, the more pronounced acidification and sugar consumption observed in cultivated caperberries are now explicitly associated with their higher initial sugar content and higher LAB counts measured during fermentation, which likely supported more active microbial metabolism. Conversely, the relatively slower

fermentation kinetics observed in wild samples are discussed in relation to their higher polyphenol levels, which may exert inhibitory effects on microbial growth, as reported in previous studies on phenolic–microbial interactions. Overall, these results suggest that the origin of caperberries (shaped by environmental, genetic, and edaphic factors) plays a key role in determining both the initial substrate composition and the structure of the indigenous microbiota, which in turn influence fermentation dynamics.

Generally, the decrease in all microbiota counts during the fermentation process could be attributed to changes in physicochemical parameters (low pH values and high levels of free acidity) and to various factors such as the depletion of nutrients (mainly sugars), high accumulation of polyphenols, and their hydrolysis products. In fact, the polyphenols from capers [33–35] and their derivatives, namely isothiocyanate [36], are known for their antimicrobial properties against bacteria and Y&M. Although no direct assessment of shelf life, environmental impact, or comparison with conventional preservation methods performed, these findings may help to prolong the shelf life of the product and maintain the stability of the fermentation environment, which aligns with sustainable preservation strategies by reducing food alteration and enhancing product stability without synthetic additives.

The initial increase of vitamin C in brine obtained on the eighth day of the process (1.251 mg/100 g) can be explained by its release from fruits into brines favored by the enzymatic degradation of the cell walls of fruits. While the subsequent decrease in vitamin C content to 0.3 mg/100 g at the end of the process is primarily due to several degradation mechanisms. It could be related to its degradation by chemical and enzymatic oxidation [37]. Indeed, ascorbic acid is highly susceptible to oxidation, particularly in the presence of dissolved oxygen, where it is converted to dehydroascorbic acid and then degraded into inactive compounds. Furthermore, microbial activity can indirectly contribute to vitamin C degradation through its metabolic activity (progressive acidification of the medium), altering the redox conditions, which influence vitamin C stability and accelerate its oxidation. A similar result of vitamin C content (0.4 mg/100 g FW) was reported previously in Moroccan commercial caperberries [38]. Moreover, our results demonstrating the significant difference ($p < 0.05$) in vitamin C when comparing wild caperberries (0.633 mg/100 g) to cultivated ones (0.56 mg/100 g) (Table 1) agree with those observed for wild apple fruits and cultivated ones [39]. This finding encourages the consumption of wild caperberries due to their high vitamin C in order to increase their intake.

The accumulation of TPC (243.99 mgGAE/L as the mean on the 15th day) in brines can be primarily attributed to their release from caperberries into the brine. We hypothesize that this initial increase is mainly governed by mass transfer phenomena, including osmotic exchanges and the progressive weakening of cell wall structures under saline conditions, which facilitate the diffusion of soluble phenolic compounds. In contrast, the subsequent decrease observed during fermentation (214.35 mg GAE/L) is unlikely to result solely from physical loss but rather from biochemical transformations. We propose that this decline is driven by microbial and enzymatic activities, including the action of oxidative enzymes (e.g., polyphenol oxidases) and the metabolic activity of LAB, which may contribute indirectly to phenolic modification through enzymatic activities (Esterases and Decarboxylases). These processes may lead to the formation of less detectable or less reactive derivatives, thereby contributing to the apparent reduction in total phenolic content. Overall, the observed trend reflects a balance between the initial release of phenolic compounds and their subsequent biotransformation during fermentation. In fact, it has been reported that polyphenols from caperberries are metabolized by microbiota present during fermentation, and glucocapparin, the principal phenolic responsible for the bitterness of capers [28], undergoes degradation into methyl isothiocyanate [18,21,40]. Furthermore, as reported for cruciferous vegetables, a complete transformation of glucosinolates and polyphenols occurs during the lactic fermentation

process [41]. On the other hand, the significant differences in TPC (181.08 mgGAE/L in wild caperberries and 166.87 mgGAE/L in cultivated ones) are reported for the first time and are in agreement with several works conducted on other berries and fruit species in which higher TPC was found in wild species than in cultivated ones [31,42–45]. Our finding could be attributed to the significant differences previously revealed, based on quantitative and qualitative parameters, between the two origins of caperberries [6], which could be related in turn to various factors such as geographical coordinates, climatic, edaphic, topographic, and solar exposure [46,47]. The higher polyphenol content in the wild provenance highlights the potential value of wild plant resources in sustainable food systems, a thing that justifies and supports their conservation and valorization.

The DPPH assay evaluates the ability of antioxidants to scavenge stable free radicals through hydrogen atom transfer (HAT) and, to some extent, single electron transfer (SET) mechanisms. Meanwhile, the FRAP assay measures the reducing power of the sample, specifically its capacity to reduce Fe^{3+} to Fe^{2+} via an electron transfer (SET) mechanism under acidic conditions. The maximal antioxidant activity (71% and 875 µgAAE/mL of DPPH and FRAP assays, respectively) obtained on the 15th day of the process indicates the high ability of the sample brine to scavenge DPPH and FRAP radicals. This finding agrees well with [18,48,49], reporting the increase of antioxidant activity values with fermentation progress. This maximal antioxidant activity obtained during this period of the process coincides simultaneously with a maximal accumulation of TPC in brine, which explains their potential antioxidants, as previously reported as the main cause of the antioxidant activity of capers [50]. The enhancement of antioxidant capacity through fermentation is of important interest since it can contribute to the development of functional foods with added health value, supporting sustainable preventive health strategies. However, the progressive decrease of antioxidant activity at the end of the process (47%, 520 µgAAE/mL of DPPH and FRAP assays, respectively) could be related to a decrease in polyphenol content due to their metabolism by the enzymatic transformations from autochthonous microbiota during the fermentation process. Furthermore, the results showing significant differences in antioxidant activity of brine samples based on DPPH and FRAP observed during the time of the fermentation process are in agreement with several studies reporting the antioxidant activity of caper fruits of *C. spinosa* [38,50–54]. Ref. [18] evaluated the antioxidant activities of caper fruits before and after the fermentation process. Moreover, the found results of significant and positive correlations of TPC and DPPH ($r = 0.823$ **) and FRAP ($r = 0.907$ ***) and between DPPH AI% and FRAP ($r = 0.889$ ***), are similar to previously reported ones [50,54].

The results of significant differences in antioxidant activity based on DPPH and FRAP assays due to the effect of provenance could be related to the highest TPC found in the brine of wild caperberries compared to that in cultivated ones. Similar results were observed in wild red raspberries, strawberries, and blackberries compared to cultivated ones [44,45,55]. Moreover, the antioxidant activity was reported as much higher in wild blueberries than in cultivated ones and was attributed more to the total phenolic [42,43]. On the other hand, it was reported that hydrophilic constituents contribute more toward the antioxidant properties of fresh caperberries [54], which justifies the antioxidant potential obtained in the caperberries' brine of our study. These findings indicate clearly that brines of fermenting caperberries possess an important antioxidant activity due to their high TPC and their derivatives accumulated during the fermentation process.

Although this study provides a comprehensive analysis of fermentation dynamics, some limitations should be acknowledged. Advanced analytical techniques, such as high-performance liquid chromatography for phenolic profiling and culture-independent microbiological approaches, could provide deeper insights into the molecular and microbial

diversity. Future studies should focus on these aspects to further elucidate the mechanisms underlying fermentation.

The obtained correlations highlight key interactions during caperberry fermentation. The strong negative correlation between pH and acidity confirms the decrease in pH as acidity rises, a typical indicator of microbial activity of the LAB population, namely lactic fermentation. Conversely, free acidity negatively correlated with Enterobacteriaceae, confirming its inhibitory effect on undesirable microbiota, and consequently, it demonstrates its protective role in microbial safety. The consistent positive correlation between phenolics and antioxidant markers (FRAP and DPPH) reinforces the role of polyphenols as major contributors to antioxidant activity. The strong positive associations between acidity accumulation and biochemical parameters (polyphenols, FRAP, and DPPH) underline the essential role of fermentation in enhancing antioxidant capacity and emphasize the beneficial role of polyphenols and their existence in the fermented caperberry process. The positive correlation between polyphenols and LAB informs the tolerance and the metabolic capacity of LAB to grow in the presence of polyphenols. The negative correlation between polyphenols and Enterobacteria may explain their inhibitory effect on this undesirable microbiota. The negative correlation between chlorides and polyphenols may result from their reciprocal diffusion from brine to fruit and vice versa, and also from the transformation of polyphenols into secondary compounds. Sugars, strongly correlated with LAB and vitamin C, underline their importance as energy substrates and stabilizers. LAB, Y&M, and TAMF are highly intercorrelated ($r > 0.8$), suggesting the strong microbial diversity occurring during these processes. The rapid disappearance of Enterobacteriaceae during the early fermentation stages, the existence of Y&M at low levels, and the persistence of the LAB population and their biochemical activities at the end of the process reveal essential microbiological dynamics within this process. These interactions could illustrate how the natural fermentation operates as a self-regulated biological system that can improve food safety and functionality.

Generally, the present study demonstrates that the natural fermentation of wild and cultivated Moroccan caperberries ensures microbiological safety, improves nutritional quality, and enhances functional properties, particularly through the accumulation of polyphenols and antioxidant activity. These findings could contribute directly to the Sustainable Development Goals (SDGs) of the United Nations, principally SDG3 (Good Health and Well-Being) by promoting safe and functional foods rich in natural bioactive compounds. Indeed, the efficient spontaneous fermentation process, without the need for synthetic additives, supports environmentally friendly preservation methods aligned with SDG12 (Responsible Consumption and Production). Furthermore, the valorization of wild caperberries highlights the importance of preserving natural plant biodiversity, contributing to SDG15 (Life on Land) by reducing post-harvest losses and enhancing the added value of local agricultural products. This study also supports SDG2 (Zero Hunger) and SDG1 (No Poverty) through strengthening sustainable food systems and increasing economic opportunities for local producers.

The results of PCA for the PC1 axis reflect the gradient contrasting undesirable parameters (e.g., high pH and presence of Enterobacteriaceae) with favorable fermentation indicators such as antioxidant compounds and acidification. This opposition confirms that successful fermentation is characterized by a drop in pH and a reduction in harmful bacteria, accompanied by a rise in antioxidant capacity and polyphenol levels. PC2 suggests it captures the microbial dynamics and sugar metabolism axis. The close association of these variables implies that microbial proliferation is linked with sugar availability, which is essential to the biochemical success of fermentation. The significant negative correlations observed between polyphenols and free acidity versus chlorides and Enterobacteriaceae,

respectively, indicate that as the former increases, the undesirable microbial populations tend to disappear, which highlights the antimicrobial effects of phenolic compounds and potential acidifying in shaping microbial balance. Conversely, the positive and significant correlations found between TPC, FRAP, and DPPH confirm the close biochemical relationship between phenolic richness and antioxidant potential and their increases through the fermentation process. Furthermore, the positive correlation found between sugars, LAB, Y&M, TAMF, and vitamin C indicates that they are positively correlated and cluster together, indicating an active phase of fermentation marked by strong microbial development and efficient sugar conversion into organic acids, translated mainly by a significant installation of free acidity.

In Figure 6B, the fact that the sample at T1 (day 1) is separated from all others reflects an unfermented state and biochemical immaturity. While the intermediate stages, such as T15 and T23, are positioned near microbial and biochemical variables, they suggest an intense fermentation activity characterized by microbial proliferation, active sugar metabolism, polyphenol accumulation, and antioxidant enhancement. This period (15th to 23rd day) corresponds to the peak of total polyphenols and antioxidant activity, indicating maximal functional and nutritional value, and can be considered as the most suitable endpoint when the objective is to obtain a product with high functional quality. In contrast, the late stages (T53 and T60) are associated with low pH, indicating a stabilized acidic environment with lower microbial diversity, consistent with the completion of fermentation. Based on these results, extending the fermentation period to 53 and 60 days enhances microbiological stability and improves long-term preservation, which could be particularly recommended for storage, transport, and commercialization.

In Figure 6C (biplot), a clear grouping is observed between T15 and T23, closely aligned with sugars, LAB, TAMF, and vitamin C, representing a favorable fermentation window where biochemical transformations and microbial activity reach their peak. In contrast, T1, along with Chl, pH, and Enterobacteriaceae, forms a cluster on the opposite side, representing the early stages of fermentation, characterized by minimal microbial and biochemical progress. The shaded area indicates the optimal fermentation conditions, where LAB and yeast populations thrive, acidity increases, and antioxidant properties are maximized.

5. Conclusions

The present work showed the success of the natural fermentation process of wild and cultivated Moroccan caperberries washed initially two times (12 h for each one) and brined at 10% NaCl. Results showed that the intrinsic physicochemical factors and microbial interactions are responsible for the self-regulation of microbial dynamics in this spontaneous fermentation, through the elimination of Enterobacteria and reduction of TAMF and Y&M, leading to the dominance of LAB as the best-adapted population.

The differences in acidification kinetics between the caperberry sources may likely reflect variations in their native microbiota and substrate composition, indicating that the origin of the raw material influences LAB activity and fermentation dynamics. The fermentation period of approximately 15 to 23 days could be recommended as an optimum time to obtain improved functional properties of fermented caperberries. While longer fermentation (up to 60 days) may be preferable when extended shelf life and commercial stability are the primary objectives. The wild caperberries showed superior functional performance, reflected by improved antioxidant potential, polyphenols, and vitamin C richness, finding that consequently encourages consumers to increase their intake.

As perspectives of this work, the isolation of autochthonous LAB strains from the natural fermentation of Moroccan caperberries and their technological and probiotic characterization deserve to be studied in order to develop an adequate starter culture for

controlling the caperberries' fermentation process, with a view to producing a final product with additional beneficial properties for the consumer.

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Abbreviations

The following abbreviations are used in this manuscript:

AAE	Ascorbic Acid Equivalents
ANOVA	Analysis of Variance
Chl	Chlorides
Cu	Cultivated
DCL	Deoxycholate Lactose agar medium
DPPH	2,2-Diphenyl-1-picrylhydrazyl
FRAP	Ferric Reducing Antioxidant Power
GAE	Gallic Acid Equivalents
HAT	Hydrogen Atom Transfer
LAB	Lactic Acid Bacteria
LSD	Least Significant Difference
µM	Millimolar
mL	Millilitre
mM	Millimolar
MRS	De Man, Rogosa and Sharpe agar medium
PCA	Principal Component Analysis
PC1	Principal Component 1
PC2	Principal Component 2
PDA	Plate Potato Agar
SET	Single Electron Transfer
T	Time of process (Day)
TAMF	Total Aerobic Mesophilic Flora
TPC	Total Polyphenols Content
Vit C	Vitamin C
w/*v*	Weight per Volume
Wi	Wild
Y&M	Yeasts and Molds
%	Percentage

References

1. Pulido, R.P.; Omar, N.B.; Abriouel, H.; López, R.L.; Cañamero, M.M.; Gálvez, A. Microbiological study of lactic acid fermentation of caper berries by molecular and culture-dependent methods. *Appl. Environ. Microbiol.* **2005**, *71*, 7872–7879. [CrossRef] [PubMed]
2. Septembre-Malaterre, A.; Remize, F.; Poucheret, P. Fruits and Vegetables, as a Source of Nutritional Compounds and Phytochemicals: Changes in Bioactive Compounds During Lactic Fermentation. *Food Res. Int.* **2018**, *104*, 86–99. [CrossRef] [PubMed]
3. Benkerroum, N. Traditional Fermented Foods of North African Countries: Technology and Food Safety Challenges with Regard to Microbiological Risks. *Compr. Rev. Food Sci. Food Saf.* **2013**, *12*, 54–89. [CrossRef]
4. Abouloifa, H.; Gaamouche, S.; Ghabbour, N.; El Guerrouj, B.; Karboune, S.; Saalaoui, N.; Asehraou, A. Lactic acid bacteria from Moroccan traditional foods: Techno-functional, health-promoting, nutraceutical value and application as a starter and bio-preservative agent in the food products. *Bioresour. Technol. Rep.* **2024**, *27*, 10194. [CrossRef]
5. Benseghir-Boukhari, L.A.; Seridi, R. Le câprier, une espèce arbustive pour le développement rural durable en Algérie. *J. Medit. Geogr.* **2007**, *109*, 101–105. [CrossRef]
6. Libiad, M.; Khabbach, A.; Ennabili, A. Genotypic variation effect on quality determination of caper products collected in North-Central Morocco. *Bol. Latinoam. Caribe Plantas Med. Aromát.* **2022**, *21*, 757–770. [CrossRef]
7. El amri, N.; Errachidi, F.; Bour, A.; Chabir, R. Characterization of Moroccan raw and processed caper berries. *Mater. Today Proc.* **2019**, *13*, 841–849. [CrossRef]
8. Stefanucci, A.; Zengin, G.; Locatelli, M.; Macedonio, G.; Wang, C.K.; Novellino, E. Impact of different geographical locations on varying profile of bioactives and associated functionalities of Caper (*Capparis spinosa* L.). *Food Chem. Toxicol.* **2018**, *118*, 181–189. [CrossRef]
9. Ghabbour, I.; Ghabbour, N.; Khabbach, A.; Louahlia, S.; Hammani, K. Checklist of the Medicinal flora used by the local population in the Province of Taza (North-Eastern Morocco) through an Ethnobotanical Study. *Bot. Sci.* **2024**, *102*, 854–877. [CrossRef]
10. Ghabbour, I.; Ghabbour, N.; Khabbach, A.; Louahlia, S.; Hammani, K. New ethnobotanical know-how characterizing the medicinal flora of the province of Taza (northern Morocco): Valorization and quantification of qualitative knowledge. *Ethnobot. Res. Appl.* **2024**, *29*, 1–32. [CrossRef]
11. Gull, T.; Anwar, F.; Sultana, B.; Cervantes Alcayde, M.A.; Nouman, W. Capparis species: A potential source of bioactives and high-value components: A review. *Ind. Crops Prod.* **2015**, *67*, 81–96. [CrossRef]
12. Ghabbour, I.; Ghabbour, N.; Khabbach, A.; Louahlia, S.; Hammani, K. Ethnobotanical statistics of disease groups treated by medicinal plants used in the province of Taza (northern Morocco). *Ethnobot. Res. Appl.* **2023**, *26*, 1–23. [CrossRef]
13. Sun, Y.; Yang, T.; Wang, C. *Capparis spinosa* L. as a potential source of nutrition and its health benefits in foods: A comprehensive review of its phytochemistry, bioactivities, safety, and application. *Food Chem.* **2023**, *409*, 135258. [CrossRef] [PubMed]
14. Saadaoui, E.; Guetat, A.; Tlili, N.; El Gazzah, M.; Khaldi, A. Subspecific variability of Tunisian wild populations of *Capparis spinosa* L. *J. Med. Plant Res.* **2011**, *5*, 4339–4348.
15. Chedraoui, S.; Abi-Rizk, A.; El-Beyrouthy, M.; Chalak, L.; Ouaini, N.; Rajjou, L. *Capparis spinosa* L. in a systematic review: A xerophilous species of multi values and promising potentialities for agrosystems under the threat of global warming. *Front. Plant Sci.* **2017**, *8*, 1845. [CrossRef]
16. Arslan, D.; Ozcan, M.M. Effect of some organic acids, yoghurt, starter culture and bud sizes on the chemical properties of pickled caper buds. *J. Food Sci. Technol.* **2007**, *44*, 66–69.
17. Arslan, D.; Ünver, A.; Özcan, M. Determination of optimum fermentation quality of capers (*Capparis ovata* Desf. var. canescens) in different brine conditions. *J. Food Process. Preserv.* **2008**, *32*, 219–230. [CrossRef]
18. Jiménez-López, J.; Ruiz-Medina, A.; Ortega-Barrales, P.; Llorent-Martínez, E.J. Phytochemical profile and antioxidant activity of caper berries (*Capparis spinosa* L.): Evaluation of the influence of the fermentation process. *Food Chem.* **2018**, *250*, 54–59. [CrossRef]
19. Giuffrida, D.; Salvo, F.; Ziino, M.; Toscano, G.; Dugo, G. Initial investigation on some chemical constituents of capers (*Capparis spinosa* L.) from the island of Salina. *Ital. J. Food Sci.* **2002**, *14*, 25–31.
20. Douieb, H.; Benlemlih, M.; Errachidi, F. Improvement of the lactic acid fermentation of capers through an experimental factorial design (*Capparis spinosa* L.). *Grasas Aceites* **2010**, *61*, 398–403. [CrossRef]
21. Palomino, J.M.; del Árbol, J.T.; Benomar, N.; Abriouel, H.; Cañamero, M.M.; Gálvez, A. Application of *Lactobacillus plantarum* Lb9 as starter culture in caper berry fermentation. *LWT–Food Sci. Technol.* **2015**, *60*, 788–794. [CrossRef]
22. Pulido, R.P.; Benomar, N.; Cañamero, M.M.; Abriouel, H.; Gálvez, A. Fermentation of caper products. In *Handbook of Plant-Based Fermented Food and Beverage Technology*; CRC Press: Boca Raton, FL, USA, 2012; pp. 201–208.
23. Ghabbour, N.; Rokni, Y.; Abouloifa, H.; Bellaouchi, R.; Hasnaoui, I.; Gaamouche, S.; Houmy, N.; El Yamani, M.; Ben Salah, R.; Ktari, N.; et al. Controlled fermentation of heat-shocked, unsalted and inoculated Moroccan Picholine green olives. *Grasas Aceites* **2023**, *74*, e485. [CrossRef]
24. Dinesh, B.; Yadav, B.; Reddy, R.D.; Padma, A.S.; Sukumaran, M.K. Determination of ascorbic acid content in some Indian spices. *Int. J. Curr. Microbiol. Appl. Sci.* **2015**, *4*, 864–868.
25. Rao, B.S.; Deshpande, V. *Experimental Biochemistry: A Student Companion*; Anshan: Tunbridge Wells, UK, 2006; 302p, ISBN 9781904798514.

26. Sarikurkcu, C. Antioxidant activities of solvent extracts from endemic Cyclamen mirabile Hildebr. tubers and leaves. *Afr. J. Biotechnol.* **2011**, *10*, 831–839. [CrossRef]
27. Zengin, G.; Nithiyanantham, S.; Locatelli, M.; Ceylan, R.; Uysal, S.; Aktumsek, A. Screening of in vitro antioxidant and enzyme inhibitory activities of different extracts from two uninvestigated wild plants: *Centranthus longiflorus* subsp. *longiflorus* and *Cerinthe minor* subsp. *Auriculata*. *Eur. J. Integr. Med.* **2016**, *8*, 286–292. [CrossRef]
28. Francesca, N.; Barbera, M.; Martorana, A.; Saiano, F.; Gaglio, R.; Aponte, M.; Moschetti, G.; Settanni, L. Optimised method for the analysis of phenolic compounds from caper (*Capparis spinosa* L.) berries and monitoring of their changes during fermentation. *Food Chem.* **2016**, *196*, 1172–1179. [CrossRef]
29. Aksay, O.; Selli, S.; Kelebek, H. LC-DAD-ESI-MS/MS-based assessment of the bioactive compounds in fresh and fermented caper (*Capparis spinosa*) buds and berries. *Food Chem.* **2021**, *337*, 127959. [CrossRef]
30. Sánchez, A.H.; De Castro, A.; Rejano, L. Controlled fermentation of caperberries. *J. Food Sci.* **1992**, *57*, 675–678. [CrossRef]
31. Mikulic-Petkovsek, M.; Schmitzer, V.; Slatnar, A.; Stampar, F.; Veberic, R. Composition of sugars, organic acids, and total phenolics in 25 wild or cultivated berry species. *J. Food Sci.* **2012**, *77*, C1064–C1070. [CrossRef]
32. Ghabbour, N.; Rokni, Y.; Lamzira, Z.; Thonart, P.; Chihib, N.E.; Peres, C.; Asehraou, A. Controlled fermentation of Moroccan picholine green olives by oleuropein-degrading lactobacilli strains. *Grasas Aceites* **2016**, *67*, e138. [CrossRef]
33. Gull, T.; Sultana, B.; Bhatti, I.A.; Jamil, A. Antibacterial potential of *Capparis spinosa* and Capparis decidua extracts. *Int. J. Agric. Biol.* **2015**, *17*, 727–733. [CrossRef]
34. Sherif, M.M.; El-Shiekh, H.H.; Elaasser, M.M.; Elbadry, M. In vitro evaluation of antimicrobial and cytotoxic effects of caper (*Capparis spinosa*). *J. Appl. Sci. Res.* **2013**, *9*, 2944–2950.
35. Adwan, G.M.; Omar, G.I. Evaluation of antimicrobial activity and genotoxic potential of *Capparis spinosa* (L.) plant extracts. *Microbiol. Res. J. Int.* **2021**, *31*, 48–57. [CrossRef]
36. Drobnica, L.; Zemanova, M.; Nemec, P.; Antos, K.; Kristian, P.; Stullerova, A.; Knoppová, V.; Nemec, P., Jr. Antifungal activity of isothiocyanates and related compounds: I. Naturally Occurring Isothiocyanates and Their Analogues. *Appl. Microbiol.* **1967**, *15*, 701–709. [CrossRef]
37. Kaprasob, R.; Kerdchoechuen, O.; Laohakunjit, N.; Thumthanaruk, B.; Shetty, K. Changes in physico-chemical, astringency, volatile compounds and antioxidant activity of fresh and concentrated cashew apple juice fermented with *Lactobacillus plantarum*. *J. Food Sci. Technol.* **2018**, *55*, 3979–3990. [CrossRef] [PubMed]
38. Tlili, N.; Elfalleh, W.; Saadaoui, E.; Khaldi, A.; Triki, S.; Nasri, N. The caper (*Capparis* L.): Ethnopharmacology, phytochemical and pharmacological properties. *Fitoterapia* **2011**, *82*, 93–101. [CrossRef]
39. Fang, T.; Zhen, Q.; Liao, L.; Owiti, A.; Zhao, L.; Korban, S. Variation of ascorbic acid concentration in fruits of cultivated and wild apples. *Food Chem.* **2017**, *225*, 132–137. [CrossRef]
40. Kulisic-Bilusic, T.; Schmöller, I.; Schnäbele, K.; Siracusa, L.; Ruberto, G. The anticarcinogenic potential of essential oil and aqueous infusion from caper (*Capparis spinosa* L.). *Food Chem.* **2012**, *132*, 261–267. [CrossRef]
41. Šalić, A.; Šamec, D. Changes in the content of glucosinolates, polyphenols and carotenoids during lactic-acid fermentation of cruciferous vege-tables: A mini review. *Food Chem.* **2022**, *16*, 100457. [CrossRef]
42. Giovanelli, G.; Buratti, S. Comparison of polyphenolic composition and antioxidant activity of wild Italian blueberries and some cultivated varieties. *Food Chem.* **2009**, *112*, 903–908. [CrossRef]
43. Bunea, A.; Rugina, O.D.; Pintea, A.M.; Sconţa, Z.; Bunea, C.I.; Socaciu, C. Comparative polyphenolic content and antioxidant activities of some wild and cultivated blueberries from Romania. *Not. Bot. Horti Agrobot.* **2011**, *39*, 70–76. [CrossRef]
44. Guerrero, C.J.; Ciampi, P.L.; Castilla, C.A.; Medel, S.F.; Schalchli, S.H.; Hormazabal, U.E.; Bensch, E.T.; Alberdi, M.L. Antioxidant capacity, anthocyanins, and total phenols of wild and cultivated berries in Chile. *Chil. J. Agric. Res.* **2011**, *70*, 537–544. [CrossRef]
45. Milivojevic, J.; Maksimovic, V.; Nikolic, M.; Bogdanovic, J.; Maletic, R.; Milatovic, D. Chemical and antioxidant properties of cultivated and wild Fragaria and Rubus berries. *J. Food Qual.* **2011**, *34*, 1–9. [CrossRef]
46. Conforti, F.; Marcotullio, M.C.; Menichini, F.; Statti, G.A.; Vannutelli, L.; Burini, G.; Menichini, F.; Curini, M. The influence of collection zone on glucosinolates, polyphenols and flavonoids contents and biological profiles of *Capparis sicula* ssp. *sicula*. *Food Sci. Technol. Int.* **2011**, *12*, 87–97. [CrossRef]
47. Conforti, F.; Modesto, S.; Menichini, F.; Statti, G.A.; Uzunov, D.; Solimene, U.; Duez, P.; Menichini, F. Correlation between environmental factors, chemical composition, and antioxidative properties of caper species growing wild in Calabria (South Italy). *Chem. Biodivers.* **2011**, *8*, 518–531. [CrossRef]
48. Hur, S.J.; Lee, S.Y.; Kim, Y.C.; Choi, I.; Kim, G.B. Effect of fermentation on the antioxidant activity in plant-based foods. *Food Chem.* **2014**, *160*, 346–356. [CrossRef]
49. Özcan, M.M.; Ahmed, I.A.M.; Juhaimi, F.A.; Uslu, N.; Osman, M.A.; Gassem, M.A. The influence of fermentation and bud sizes on antioxidant activity and bioactive compounds of three different size buds of *Capparis ovata* Desf. var. *canescens* plant. *J. Food Sci. Technol.* **2020**, *57*, 2705–2712. [CrossRef]

50. Bhoyar, M.S.; Mishra, G.P.; Naik, P.K.; Singh, S.B. Evaluation of antioxidant capacities and total polyphenols in various edible parts of *Capparis spinosa* L. collected from trans-Himalayas. *Def. Life Sci. J.* **2018**, *3*, 140–145. [CrossRef]
51. Tesoriere, L.; Butera, D.; Gentile, C.; Livrea, M.A. Bioactive components of caper (*Capparis spinosa* L.) from Sicily and antioxidant effects in a red meat simulated gastric digestion. *J. Agric. Food Chem.* **2007**, *55*, 8465–8471. [CrossRef]
52. Yang, T.; Wang, C.; Liu, H.; Chou, G.; Cheng, X.; Wang, Z.A. new antioxidant compound from *Capparis spinosa*. *Pharm. Biol.* **2010**, *48*, 589–594. [CrossRef] [PubMed]
53. Aliyazicioglu, R.; Eyupoglu, O.E.; Sahin, H.; Yildiz, O.; Baltas, N. Phenolic components, antioxidant activity, and mineral analysis of *Capparis spinosa* L. *Afr. J. Biotechnol.* **2013**, *12*, 6643–6649. [CrossRef]
54. Allaith, A.A.A. Assessment of the antioxidant properties of the caper fruit (*Capparis spinosa* L.) from Bahrain. *J. Assoc. Arab. Univ. Basic Appl. Sci.* **2016**, *19*, 1–7. [CrossRef]
55. Çekiç, Ç.; Özgen, M. Comparison of antioxidant capacity and phytochemical properties of wild and cultivated red raspberries (*Rubus idaeus* L.). *J. Food Compos. Anal.* **2010**, *23*, 540–544. [CrossRef]

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