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Effect of Natural Marination Based on Apple Vinegar and Acid Whey on Volatile and Sensory Profile, Safety, and Physicochemical Properties of Raw Fermented Beef Hams

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Abstract: Consumers appreciate fermented meat products due to their nutritional value and unique taste. Fermented fruit vinegar used traditionally to preserve food is characterized by a high content of nutrients and bioactive ingredients. Acid whey has antioxidant and antibacterial properties and can provide probiotic lactic acid bacteria (LAB). Three variants of the fermented beef hams were produced: AW—1.5% salt and 5% acid whey, A—1.5% salt and 5% apple vinegar, and C—1.5% salt. The effect of natural marination, based on apple vinegar and acid whey on volatiles, physicochemical and microbiological parameters, and sensory quality of the raw fermented beef hams was assessed. The highest pH and the lowest oxidation–reduction potential value (ORP) was found for A hams after production and after storage ($p < 0.05$). AW hams had the highest Thiobarbituric Acid-Reactive Substances (TBARS) value after production (6.07 mg MDA/kg) and after 8 months of storage (6.12 mg MDA/kg) ($p < 0.05$). The AW and A hams showed moderate overall sensory quality after production and after storage (above 5 c.u.). Both treatments modify the formation and stability of volatile compounds, not affecting the overall quality. The number of LAB in raw fermented beef hams with acid whey and apple vinegar was high (approximately 7 log CFU/g) after 3 months, and this decreased after 8 months of storage to 6.24 and 5.83 log CFU/g, respectively, for AW and A treatment. Among sixty volatile compounds, an abundance of aldehydes, carboxylic acids, esters, and alcohols dominated, which contributed to the formation of aroma attributes of beef hams. This study demonstrates that apple vinegar and acid whey can be used for the production of microbiologically safe fermented beef hams with good sensory quality.

Keywords: vinegar; acid whey; lactic acid bacteria; volatiles; raw fermented meat

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1. Introduction

For centuries, meat fermentation has been a common form of preservation that enables storage, ensuring the meat's safety by reducing or eliminating spoilage bacteria occurrence

and the risk of contamination by pathogenic microorganisms [1,2]. Fermented meat products are characterized by improved nutritional values and unique taste and sensory properties. Microorganisms, mainly lactic acid bacteria (LAB), are present as natural microbiota in the fermented meat or are added as a starter culture. LAB are acid-tolerant bacteria. The main metabolite is lactic acid, which is responsible for the acidifications of fermented meat. The acid counteracts against spoilage and pathogenic microorganisms, contributing to raw meat preservation. LAB also produce other antimicrobial chemicals such as bacteriocins (i.e., nisin, pediocin), other organic acids (i.e., acetic and propionic acid), and small molecules (i.e., diacetyl, hydrogen peroxide, carbon dioxide, exopolysaccharides) [3]. Food products containing at least 10^6 CFU of a viable probiotic LAB strain are considered as a functional food and are preferred by consumers. In addition to biopreservation and consumer health benefits, LAB also influence the proteins, lipids, and carbohydrates, forming a unique meat taste [4–6]. During fermentation processes, microbial enzymes contribute to the hydrolysis of myofibrillar proteins, increasing the level of free amino acids as well as small-molecular-weight proteins. The proteolytic activity of LAB is strain-dependent [7]. According to Liu et al. (2022), in Jinhua hams, the activity of LAB aminotransferases leads to the elevation of branched aldehydes, acids, and alcohols, influencing meat product aroma [8]. Taste and flavors are important indicators for quality assessment. Flavor compounds are formed by various chemical and biochemical reactions such as protein hydrolysis, carbohydrate breakdown, and lipid oxidation [9]. The estimations of dry-cured ham production and storage showed that, in different stages of the ripening process, approximately 1000 metabolites were detected [10]. The metabolite profiles change with the storage time and are influenced by the type of treatment. The ripening of fresh, unsalted, and dried meat generated other profiles than salted samples [10]. Many of the volatile compounds present in dry-ripened hams have high odor thresholds or are not odor-active compounds and, therefore, do not contribute to aroma formation [11]. Major odor-active volatile compound groups shaping the flavor profile of dry-cured ham include carboxylic acids, esters, aldehydes, ketones, furans, pyridines, pyrazines, and thiazoles [12]. According to Flores et al., compounds of important impact on dry-cured ham aroma include hydrogen sulfide, methanethiol, 2-methyl-3-furanthiol and 2-methyl-3-methyl-dithiofuran, pyrrole, 2-acetyl-1-pyrroline and 2-pro pionyl-1-pyrrolinemethylpyrazine, dimethylpyrazine, tetramethylpyrazine and 3-isopropyl-2-methoxypyrazine, and 2-acetyl- 2-thiazoline [13].

Nowadays, more attention is paid to organic chemical- preservative- free meat products. Data from the literature confirm the effectiveness of acid whey as an ingredient of meat products with antioxidant, antibacterial, and potentially health-promoting properties. Acid whey is a by-product of cottage cheese production, which contains approximately 93% water, 4.8% lactose, 0.8% protein, and 0.5% fat [14]. Acid whey could be used in the production of ripened meat products [15–18]. Acid whey is a source of potentially probiotic lactic acid bacteria and potentially health-promoting ingredients. LAB can hydrolyze whey proteins, providing bioactive compounds. During the souring of milk, in whey, numerous metabolites of lactic acid bacterial strains are formed, i.e., with bactericidal and bacteriostatic properties (e.g., lactic acid and bacteriocins) [19,20]. It can positively influence the oxidative changes, physicochemical parameters, and microbiological quality of raw-ripened meat products. In the study of Karwowska and Kononiuk, the acid whey addition decreased the pH of dry-fermented nitrite-free beef sausage, which contributed to the microbiological safety of sausages [15]. Amino acids contained in acid whey, such as methionine and cysteine, have anti-cancer effects [21,22]. The whey-based fermented food products are considered to provide positive immunomodulation in human health [23].

Vinegar has been commonly used for centuries as a food preservative [24]. Vinegar from natural fermentation is characterized by a high content of nutrients and bioactive

ingredients, and has antioxidant effects [25]. It can be used in the technological processing of meat and has a positive impact on sensory properties, color, tenderness, microbiological safety, and the shelf life of meat products [26]. Wang et al. (2023) detected 64 volatile flavor components during vinegar fermentation [27]. Naturally fermented apple vinegar is used in the technological processing of meat. Due to bioactive components (organic acid, polyphenols, vitamins, minerals), vinegar can be considered as a potential postbiotic that can support LAB growth. Two major groups of apple vinegar bacteria are acetic acid and lactic acid bacteria [28–30]. Acetic and lactic acid are two prominent organic acids of vinegar. They can lower the pH of fermented food matrices [27]. Antimicrobial compounds produced by acetic acid and LAB along with organic acids and phenolic and flavonoid compounds have antimicrobial activity that ensure the microbiological safety of marinated food [31]. Acid vinegar limits the development of unfavorable microbiota, including *Listeria monocytogenes*, *Staphylococcus aureus*, *Salmonella Typhimurium*, and *Escherichia coli, Bacillus cereus, Bacillus subtilis,* and *Enterococcus faecalis* [31,32].

Additionally, the biological ingredients of fruit vinegar contribute to the health-promoting effect of meat products on the human body [33–35]. The application of natural preservatives enables the replacement or reduces the use of synthetic preservatives, resulting in positive effects that are appreciated and accepted by customers [36].

In meat processing, nitrate and nitrite commonly inhibit the growth of microorganisms, delay rancidity, and produce a pinkish-red colorand improve fflavor. However, consuming processed meat cured with nitrate and nitrite involves serious health risks [37]. Nitrite-cured meat is regarded as a Group 1 carcinogen by IARC [38]. Thus, effective methods for replacing nitrite or nitrate in processed meat are needed.

The attractiveness of fermented meat products for consumers and the food industry can be achieved through reducing or replacing nitrite; unique sensory qualities and the potential health-promoting effect of LAB can be increased [7,39,40]. Thus, this study aimed to evaluate the effect of using acid whey or apple vinegar as natural alternative additives in raw fermented beef hams. The idea was to use natural ingredients instead of chemical additives in innovative raw fermented beef hams without adding curing salt. Specifically, this study focused on evaluating the effect of the proposed natural marination process on the physicochemical, microbiological quality, and volatile and sensory profiles of the raw fermented beef hams.

2. Materials and Methods

2.1. Organic Acid Whey

The acid whey, obtained from the production of organic cottage cheese, was manufactured in a dairy plant located in Dukla, Poland. The mean pH value was 4.85 and the oxidation–reduction potential (ORP) value was 425.67. The color parameters of acid whey were L* = 35.09, a* = −5.63, and b* = 4.39. The number of lactic acid bacteria was approximately 7.05 log CFU/mL. There were none of the following pathogens: *L. monocytogenes* or *Salmonella* spp.

2.2. Apple Vinegar

Cold-pressed apple juice (variety Champion), sourced from farms, was used as the raw material to produce vinegar. The apple juice was prepared and supplied by four farms in the Łódź province. In the first stage, apple wine was obtained as a result of anaerobic fermentation (25 °C) of the apple juice using Tokay wine yeast. Then, acetic acid was biosynthesized at 30 °C from the above apple wine using *Acetobacter pasteurianus* O4 and *Acetobacter pasteurianus* MW3 (respectively, KKP 674; GenBank accession OM200034 and KKP 2997; GenBank acc. no212983), which came from the Institute of Agricultural and

Food Biotechnology State Research Institute's own microbial culture collection (Warsaw, Poland) [41].

The values of pH and ORP of the apple vinegar were 3.31 and 469.67, respectively. The strength of the vinegar was approximately 3–4 g of acetic acid/100 mL, and alcohol content was up to 1.3%. The acetic acid bacteria counts were approximately 6 log CFU/mL. The apple vinegar was characterized by a light-yellow color, a typical fruity, aromatic, and sour apple vinegar odor, acetic, and a fruity flavor [42].

2.3. Manufacture of Raw Fermented Beef Hams

For the production of raw fermented beef hams, m. *semimembranosus* was selected. The muscles were obtained from 18 beef cattle. Eighteen pieces of muscle without skin and fat were used. The weight of each individual meat element varied from 1200 g to 1500 g. The meat material was excised from carcasses chilled at 2 °C 48 h after *post mortem*. Meat elements were randomly divided into three experimental variants (six pieces in each). Under industrial conditions three different treatments of the fermented beef hams were produced: the AW treatment with 1.5% salt (*w*/*w*) and 5% organic unpasteurized acid whey (*w*/*w*); the A treatment with 1.5% salt (*w*/*w*) and 5% cold apple vinegar (*w*/*w*), and the C treatment with 1.5% salt (*w*/*w*) and cold water. The salt used was Kłodawa rock salt, NaCl content—97% min., manufacturer: "Kłodawa", Kłodowa, Poland. The amount of vinegar and acid whey added was determined according to previous microbiological, physicochemical, and sensory tests. The following raw material preparation process was used: beef muscles were properly processed and divided into pieces. Depending on the experimental variant, the raw hams were salted manually, and then vinegar or acid whey was rubbed by hand into the raw material. Water was used to maintain mass consistency in variants with no added vinegar or acid whey. The hams were stored for 48 h in a cooling chamber (temperature of 4 °C), and then glucose (5 g/kg) was applied separately to the raw meat material by hand. The glucose used was Glucose anhydrous, pure, manufacturer: Chempur, Piekary Śląskie, Poland. The hams were then hung on smoking sticks in a drying chamber for the ripening process (temperature 15–17 °C, relative humidity of 75–80%) for three days. Then, cold smoking was carried out (temperature 30–35 °C) for 1 h. The ripening process was continued for 4 weeks until the hams lost about 45% of their mass. After the process, each raw fermented ham was vacuum-packed in polyethylene foil bags and stored at 4–6 °C. The products were analyzed after production (time 0) and after 3 (time 1) and 8 (time 2) months of cold storage (4–6 °C). Two separate production batches were prepared in the meat processing plant located in Dukla, Poland. There were four biological replicates for treatment (n = 4).

2.4. pH Value, TBARS (Thiobarbituric Acid-Reactive Substances) Index, and Redox Potential Value

The pH value was determined with a digital Delta 350 pH meter (Mettler Toledo, Schwerzenbach, Switzerland) with an In Lab Cool electrode (Mettler Toledo, Greifensee, Switzerland) according to ISO 2917:1999 [43]. The blender (MSM 66120, BSH Hausgeräte GmbH, Munich, Germany) was used to homogenize the mixture of 10 g of meat sample in 50 mL of water. The pH meter was calibrated prior to analysis using 2.0, 4.0, 7.0, and 10.0 buffer solutions. Measurements were carried out at 20 °C.

Absorbance at the wavelength of 532 nm was measured to determine TBARS index using a U—2900 spectrophotometer (Hitachi, Tokyo, Japan), following the method of Pikul et al. (1989) [44]. Measurements were carried out at 20 °C. The value of TBARS was expressed as mg malondialdehyde/kg of meat and quantified according to the following formula: TBARS (mg MDA kg^{-1} sample) = 5.5 × absorbance.

The oxidation–reduction potential was measured with a sevenCompactTM S220 with an InLab Redox electrode (Mettler—Toledo, Greifensee, Switzerland) following Okoń et al.'s (2021) methodology [45]. The ORP value is expressed in mV. Each analysis was conducted with four replicates.

2.5. Analysis of Microbiological Quality

The meat sample in the amount of 10 or 25 g was diluted appropriately with sterile buffered peptone water (Bio—Rad, Hercules, CA, USA) to obtain the first dilution in the proportion of 1:10 (*w*/*v*). After homogenization for 90 s in the stomacher, the suspension was decimally diluted, and proper dilutions were plated on appropriate culture media. The number of lactic acid bacteria was determined according to ISO 15214:1998 on MRS agar (de-Man Rogosa and Sharpe agar, Merck, Germany) [46]. Nutrient agar (LabM, Heywood, UK) was used for determination of total viable count (TVC) according to ISO 4833—1:2013 [47]. The *Enterobacteriaceae* family (ENT) was enumerated according to ISO 21528—2:2017 on the Mac Conkey agar, Merck, Germany [48]. The number of coagulase-positive staphylococci (*Staphylococcus aureus* and other species) (SA) was determined on Baird-Parker agar with egg yolk tellurite (Merc, Germany) according to ISO 6888—1:2021 [49]. The presence of *Salmonella* spp. (SAL) was detected on XLD agar (LabM, Heywood, UK) according to ISO 6579—1:2017 [50]; the presence of *Listeria monocytogenes* (LIST) was detected on ALOA agar (*Listeria* selective agar acc. to Ottaviani and Agosti, Bio—Rad, USA) and PALCAM agar (Polymyxin Acriflavine Lithium—chloride Ceftazidime Aesculin—Mannitol agar, LabM, UK) according to ISO 11290—1:2017 [51]. The results were expressed as decimal logarithm (log CFU/g) of meat product.

2.6. Fatty Acid Composition

Fatty acids were analyzed by gas chromatography using HP/Agilent 6890 II—FID (Hewlett—Packard; Palo Alto, CA, USA) equipped with a flame ionization detector and a highly polarized column with BPX 70 phase according to ISO 12966—1:2014 [52]. The results were expressed in % of total fatty acids [52]. Peak identification was made by comparing the retention times with commercial standards (Supelco, 37 Component FAME CRM47885; Supelco, Bellefonte, PA, USA).

2.7. Analysis of Volatile Compound Profile

Solid-phase microextraction (SPME) before gas chromatography coupled with mass spectrometry (GC/MS) was applied to determine volatile compounds (VCs) in meat samples. For SPME extraction of VCs from 5 g of sample, a triple-coated fiber DVB/CAR/PDMS (divinylbenzene/carboxy/polydimethylsiloxane; 10 mm length, 50/30 mm thickness, Supelco, Bellefonte, PA, USA) was used. The extraction was achieved at 40 °C by exposing SPME fiber in the sample's headspace at 40 °C for 40 min. The details about conditions of VC extraction are described previously [45,46]. Separation was performed on GC/MS (6890N GC, 5975 MS Agilent Technologies, Santa Clara, CA, USA) with HP—5MS column (30 m × 0.25 mm × 0.25 µm film thickness, 5%—diphenyl—95%—polydimethylsiloxan; Agilent, Santa Clara, CA, USA) and run in splitless mode. Carrier gas was He at a flow rate of 0.9 mL/min. The details of the temperature oven program are described in previous papers [45,46]. The MS was set to electronic impact mode (EI) 70 eV with the source temperature at 230 °C and an ion scanning range of 33–350 *m*/*z* (amu). Volatile compounds were identified by comparing the Kovats' retention indexes (K.I.) and mass spectrum with those reported in the NIST Mass Spectral Search Program (NIST.08 and Wiley 8th Ed. Libraries) with <80% as a cutoff to match compounds. The Kovats' retention indexes (K.I.) were calculated from the retention times of C6—C20 n—alkanes (Sigma—Aldrich, Poznań, Poland).

Quantities of VCs were reported as a relative percentage of the total peak area [53,54]. The analyses were carried out in triplicates.

2.8. Quantitative Descriptive Profiling (QDP) and Trained Panel Evaluation

The tests were carried out on samples within 24 h of their production and after 8 months of storage. To evaluate the sensory quality of the samples, the QDP method was used (ISO 13299:2016) [55]. Experts rated the intensity of the sensory attributes on a 10 cm unstructured linear scale, with endpoints labeled "none" and "very intense", with the following exceptions: for color, dark–bright and for the texture attribute, low–high. Sensory quality was assessed by a trained team (9 assessors) with experience of from 4 to 20 years in the sensory evaluation of food products, including meat products (ISO 8586:2023) [56]. Two separate assessment sessions were performed. Sensory attributes (18) were defined and chosen. They were odor attributes (5): smoked, dried, sharp, storage, and other; attributes of color (2): color intensity and homogeneity and visible fat; texture attributes (1): juiciness; and attributes of flavor (8): smoked, dried, salty, bitter, storage, pungent, sour, and other. Based on all attribute characteristics, the assessors specified an overall sensory quality with boundary terms: low–very high for each raw fermented beef sample. Eighteen individual results were obtained. Sliced into equal size, raw fermented beef ham samples were placed into odorless, plastic, disposable boxes closed with lids marked separately with a three-digit code. The samples were randomly served for evaluation to avoid the carry-over effect (i.e., the impact of a previous sample on a subsequent one). The assessment was conducted in a room free from foreign odors, without noise, and at ambient temperature.

2.9. Statistical Analysis

The results were expressed as mean ± standard deviation (SD). The data regarding volatile compound profile were subjected to multiway analysis of variance followed by Tukey's post hoc test using Statistica 13.3 (TIBCO Software Inc., Palo Alto, CA, USA). Analyses of other physicochemical and microbiological parameters were investigated with two-way analysis of variance (ANOVA) using the application Statistica v. 13 (StatSoftPolska Sp. z o.o, Cracov, Poland) and the Fisher post hoc. The results for the sensory quality were conducted using a one-way ANOVA, and Tukey's HSD test was used to assess the differences between the hams in the intensity of the attributes. Correlation coefficients were calculated between individually assessed attributes and overall quality. PCA analyses were carried out to assess the influence of storage and treatment on the sensory quality of the beef hams. A level of $p < 0.05$ was considered as significant in all results.

3. Results and Discussion

3.1. TBARS, Oxidation–Reduction Potential (ORP), and pH Value

Lipid oxidation is the main cause of undesirable chemical and sensory changes resulting in the decreased quality and limited storage of fermented meat products. TBARS is a frequently used indicator to assess the degree of lipid oxidation. Lipid oxidation is one of the main causes of meat products' declined quality and accumulation of harmful end products. One the of most prominent end products is malondialdehyde (MDA), which reacts with thiobarbituric acid (TBA). MDA is linked to the unpleasant aroma of meat products. However, due to the low specificity of TBARS (TBA reacts with multiply compounds), it is difficult to assess the impact of TBARS value on sensory attributes. In general, despite the criticism, for many years, the TBARS index is a commonly used assay to monitor lipid oxidation [57]. In the presented study, TBARS was used to assess the oxidation effect of the studied treatment [15,58,59]. The secondary oxidation products' TBARS values were significantly ($p < 0,05$) affected by the kind of treatment and storage

time (Table 1). After production, hams treated with acid whey showed the highest TBARS index (6.07 mg MDA/kg). TBARS values found in the C and the A samples after production were significantly lower (respectively, 3.76 mg MDA/kg and 3.30 mg MDA/kg). After 8 months of storage, the highest TBARS value was also observed in AW samples (6.12 mg MDA/kg) compared to C and A variants (respectively, 3.53 mg MDA/kg and 5.03 mg MDA/kg). The ability of acid whey to inhibit lipid oxidation in fermented meat products can be ineffective [59].

Table 1. TBARS, oxidation–reduction potential (ORP), and pH values of raw fermented beef hams.

	Treatment	Storage Time		
		0	3 Months	8 Months
TBARS (mg MDA/kg of products)	C	3.76 ± 0.08^{aB}	1.71 ± 0.38^{aA}	3.53 ± 0.29^{aB}
	A	3.30 ± 0.31^{aA}	4.71 ± 0.17^{bB}	5.03 ± 0.40^{bB}
	AW	6.07 ± 0.11^{bB}	4.08 ± 0.24^{bA}	6.12 ± 0.38^{cB}
ORP (mV)	C	347.90 ± 11.01^{bB}	353.10 ± 8.83^{aB}	313.00 ± 6.52^{bA}
	A	330.45 ± 9.86^{aB}	361.45 ± 9.84^{aC}	306.50 ± 3.20^{aA}
	AW	341.15 ± 3.86^{bB}	374.10 ± 9.33^{bC}	310.50 ± 1.80^{bA}
pH	C	6.06 ± 0.04^{aB}	5.38 ± 0.04^{aA}	5.30 ± 0.07^{aA}
	A	6.35 ± 0.21^{bC}	5.30 ± 0.06^{aA}	5.62 ± 0.02^{bB}
	AW	5.90 ± 0.05^{aC}	5.55 ± 0.07^{bB}	5.25 ± 0.01^{aA}

Samples: AW—1.5% salt and 5% acid whey, A—1.5% salt and 5% apple vinegar, C—1.5% salt; means in the same row with different uppercase letters (A–C) differ significantly, means in the same column with different lowercase letters (a–c) differ significantly ($p < 0.05$), means ± standard deviations; n = 4.

The TBARS in the studied hams shows much higher values than those found by other authors for other fermented meat products. The TBARS values in Iberian dry-cured hams were below 0.60 mg MDA/kg, 1.51–1.59 mg MDA/kg in salted dry-cured turkey hams, 0.86–1.5 mg MDA/kg in Turkish pastirma, a dry-cured beef product, and below 1.6 mg MDA/kg in fermented eye beef round [13,60–62]. Refrigerated storage resulted in a significant ($p < 0.05$) decrease in TBARS value after 3 months in C and AW samples, which may suggest an inhibition in the rate of the oxidation reaction. The decrease in the value of the TBARS index may be related to the reaction of MDA with proteins [63]. In A treatment, an increase in TBARS value was observed during storage, which was statistically significant ($p < 0.05$) after 3 months. This can indicate that apple vinegar does not prevent oxidation in these hams during storage. The increase in TBARS value during storage could be due to the further oxidation of unsaturated fatty acids and oxidative transformations of primary oxidation products [64].

The acceptable value of TBARS in meat products without oxidized taste aroma should not exceed 2–2.5 mg MDA/kg [65,66]. However, higher values of the TBARS index (above 2 mg MDA/kg) do not necessarily indicate the poor quality of fermented meat products [67]. Some oxidative changes in raw-ripened meat products can positively impact the development of a desired and distinctive flavor in fermented meat products [68–71]. The literature on the TBARS index in relation to sensory attributes and the safety of beef products is infrequent. In a recent study, Kaczmarek and Muzolf-Panek used artificial neural networks to predict the value of the TBARS index in an assessment of raw beef storage, including the variants of beef enriched with plant-derived antioxidants. The authors reported the usefulness of the developed prediction model for the monitoring of oxidative beef changes; however, they failed to show a correlation between TBARS and plant-derived antioxidant activity [68].

The assessment of the ORP values demonstrated that C and AW samples were characterized by the highest ORP value both after production (347.90 and 341.15 mV) and

after 8 months of storage (313.00 and 310.50 mV). After 8 months of storage, a significant ($p < 0.05$) decrease in the redox potential value was noted in all ham samples.

The pH values of the beef hams studied during the entire study were in the range of 5.25–6.35 and were typical for raw-ripened meat products [18,21,26,72,73]. After production, apple vinegar treatment gave a higher pH than both the C and AW treatments. This can be explained by the behavior of the acetic acid bacteria. The concentration of 3–4 g of acetic acid/100 cm^3 in apple vinegar could be the threshold for acetic acid production by the used *Acetobacter* strains (*Acetobacter pasteurianus* O4 and *Acetobacter pasteurianus* MW3). When the threshold is exceeded, some acetic acid bacteria can further oxidize the produced acetic acid to CO_2 and H_2O_2 [74]. In A treatment, the number of lactic acid bacteria increased with a simultaneous decrease in pH (Tables 2 and 3). During storage, the LAB number also increased in C and AW variants of fermented hams, which resulted in a decrease in pH (Tables 2 and 3). After 8 months of refrigerated storage, a significant decrease in pH was noticed in all ham variants. The lower pH found in the AW and C treatments could have resulted from a higher number of lactic acid bacteria in these variants (Tables 2 and 3). The optimal pH for LAB growth is mildly acidic (5.5–6.0). During fermentation, acid-tolerant LAB produce organic acids such as lactic, acetic, and propionic acid, acidifying raw meat and inhibiting the growth of pathogenic and spoilage bacteria [75]. The most abundant organic acid produced by LAB is lactic acid and, therefore, has a great impact on acidity. The efficiency of lactic acid production is strain-dependent and related to the environmental factors such as temperature, pH, and nutrient availability, i.e., glucose. The end product could be only lactic acid or, in the hetero-fermented pathway, ethanol and CO_2. In comparison to lactic acid, the production of acetic acid by LAB is low, as well as its acidifying effect. But, due to greater non-dissociating proprieties, it could have higher antibacterial activity [75]. The comparison of AW and A beef hams showed that, after production, the TBRAS index was higher in the AW variant ($p < 0.5$). There were no statistical differences between A and AW after production. After 3 months of storage, A variant was characterized by lower pH ($p < 0.5$) and, after 8 months, AW had a lower pH ($p < 0.5$) (Table 1). The observed differences can be explained by the various dynamics of biochemical processes, as well as by the diversity of the bioactive compounds present in acid whey and apple vinegar.

3.2. Microbiological Analysis of the Tested Beef Hams

The total microbial count (TVC—total viable count) after production in all ham variants was at a low level (3.53–4.39 log CFU/g). After production, the TVC in the A samples was higher (4.39 log CFU/g) than in the control sample (3.89 log CFU/g) (Table 2). In studies by other authors on chicken meat and cooked marinated beef meat, a reduction in TVC of approximately 1.0 log CFU/g after the marinating process was found, [76]. This can be explained by the higher pH of the studied samples (Table 1), as well as a different type of meat, marination formulation, temperature, and time of marination [29]. The lactic acid bacteria (LAB) counts were also low and equalized (4.92–4.99 log CFU/g) (Table 2). The number of lactic acid bacteria reached the highest values after 3 months of storage in all beef ham samples and remained high until the end of the storage period. After production and during the storage of beef hams, there were no significant differences of LAB count in C, A, and AW variants. This indicates the possibility of beef autochthonous LAB strain predominance. This should be confirmed in further bacterial strain typing studies. According to Zhang et al. (2020), LAB levels in fermentation processes are dynamic; they decrease throughout fermentation [77]. LAB are pivotal microorganisms of fermented meat products. Unique bacterial enzymes (esterase, protease, and peroxidase), along with endogenous meat enzymes, decompose proteins, fats, and carbohydrates, producing flavor compounds

and shaping the flavor of the final fermented meat product [78]. Moreover, LAB have antioxidant and antimicrobial activity, contributing to the extended shelf life of fermented meat [3]. LAB degrade nitrates, reducing potentially carcinogenic N—nitrosamines in meat products [79].

Table 2. Microbiological quality of studied hams.

Parameter	Treatment	Storage Time		
		0	3 Months	8 Months
LAB (log CFU/g)	C	4.99 ± 0.01^{Aa}	7.15 ± 0.01^{Ba}	6.25 ± 0.09^{Ca}
	A	4.92 ± 0.01^{Aa}	7.48 ± 0.03^{Bb}	5.83 ± 0.03^{Cb}
	AW	4.97 ± 0.20^{Aa}	6.45 ± 0.07^{Bc}	6.24 ± 0.01^{Ca}
TVC (log CFU/g)	C	3.89 ± 0.01^{Ab}	7.42 ± 0.20^{Ba}	7.13 ± 0.03^{Ca}
	A	4.39 ± 0.01^{Ab}	7.31 ± 0.02^{Ba}	6.27 ± 0.10^{Cb}
	AW	3.53 ± 0.02^{Ac}	6.45 ± 0.07^{Bb}	7.36 ± 0.03^{Cc}
ENT (log CFU/g)	C	<1.00	<1.00	<1.00
	A	<1.00	<1.00	<1.00
	AW	<1.00	<1.00	<1.00
E. COLI (log CFU/g)	C	<1.00	<1.00	<1.00
	A	<1.00	<1.00	<1.00
	AW	<1.00	<1.00	<1.00
SA (log CFU/g)	C	<1.00	<1.00	<1.00
	A	<1.00	<1.00	<1.00
	AW	<1.00	<1.00	<1.00
SAL	C	nd	nd	nd
	A	nd	nd	nd
	AW	nd	nd	nd
LIST	C	nd	nd	nd
	A	nd	nd	nd
	AW	nd	nd	nd

Sample: AW—1.5% salt and 5% acid whey, A—1.5% salt and 5% apple vinegar, C—1.5% salt; LAB—lactic acid bacteria, TVC—total viable count; ENT—bacteria from the *Enterobacteriaceae* family, E. COLI—*Escherichia coli*, SA—coagulase-positive staphylococci (*Staphylococcus aureus* and other species), SAL—*Salmonella* spp., LIST—*Listeria* spp. including *L. monocytogenes*; <1.00—counts below the detection limit of the plating method; nd—not detected in 25 g of product; means in the same row with different uppercase letters (A–C) differ significantly, means in the same column with different lowercase letters (a–c) differ significantly ($p < 0.05$); means ± standard deviations; n = 4.

The strategy of replacement chemical preservatives, including nitrate and nitrite, should consider the microbiological safety of fermented meat products. In nitrate-free products, the potential microbiological hazard should be confronted by potential chemical risk. *Enterobacteriaceae*, including *Escherichia coli*, were below the detection level of the method used in all variants of hams after the fermentation process (<1.00 log CFU/g). *Staphylococcus aureus* was also not observed in all tested hams. This proves that the ham technology process was properly carried out in terms of microbiological safety. The tested hams were free from pathogens *Salmonella* and *Listeria* spp. (including *L. monocytogenes*) after production and after storage. The amount of tested natural preservatives were appropriate for the microbiological safety of the studied beef hams. However, further microbiological assessments are needed for the industrial implementation of this technology. In the design of studies on the variety of available natural preservatives, its changeable content should be considered, as well as the possible multiple interactions with the food matrix. In this regard, the use of chemical preservatives is more predictable [40].

Table 3. Fatty acid composition (% of total fatty acids).

Treatment Storage Time	C		A		AW	
	Fresh 0 Months	**8 Months**	**Fresh 0 Months**	**8 Months**	**Fresh 0 Months**	**8 Months**
SFA	41.95 ± 0.05 ^{aA}	48.10 ± 0.50 ^{bB}	47.00 ± 0.20 ^{bB}	43.00 ± 1.40 ^{aA}	45.20 ± 0.20 ^{bA}	44.90 ± 0.30 ^{aA}
MUFA	48.50 ± 0.10 ^{bB}	46.00 ± 0.20 ^{aA}	45.60 ± 0.10 ^{aA}	48.60 ± 0.70 ^{bB}	49.25 ± 0.25 ^{bB}	47.30 ± 0.40 ^{aA}
PUFA	9.55 ± 0.05 ^{bB}	5.85 ± 0.35 ^{aA}	7.35 ± 0.05 ^{aA}	8.40 ± 2.10 ^{aA}	5.40 ± 0.35 ^{aA}	7.80 ± 0.10 ^{aB}
n-3	2.50 ± 0.10 ^{bB}	1.45 ± 0.05 ^{aA}	2.55 ± 0.05 ^{bB}	1.50 ± 0.40 ^{aA}	1.70 ± 10 ^{aA}	1.95 ± 0.05 ^{aA}
n-6	6.95 ± 0.05 ^{bB}	4.30 ± 0.30 ^{aA}	4.70 ± 0.10 ^{aA}	6.80 ± 1.70 ^{bA}	3.60± 0.30 ^{aA}	5.85 ± 0.15 ^{bB}
n6/n3	2.79 ± 0.13 ^{bA}	2.96 ± 0.10 ^{aA}	1.84 0.08 ^{aA}	4.56 ± 0.08 ^{bB}	2.11 ± 0.05 ^{aA}	3.00 ± 0.15 ^{aB}

Sample: AW—1.5% salt and 5% acid whey, A—1.5% salt and 5% apple vinegar, C—1.5% salt; SFA—the sum of the saturated fatty acids; MUFA—the sum of the monounsaturated fatty acids; PUFA—the sum of the polyunsaturated fatty acids; n-3—the sum of the n-3 fatty acids; n-6—the sum of the n-6 fatty acids; means in the same row with different uppercase letters (A, B) differ significantly, means in the same column with different lowercase letters (a, b) differ significantly ($p < 0.05$), means ± standard deviations; n = 4.

3.3. Fatty Acid Composition

In the studied hams, a low content of polyunsaturated fatty acids (PUFA) was characterized from 5.40% to 9.55% after production (Table 3). The highest PUFA content was in the control hams. After 8 months of storage, the sum of PUFA decreased in the control hams, while it did not change in the hams with apple vinegar, and it increased in the hams with acid whey. PUFAs are particularly susceptible to oxidation [80,81]. This can be related to the results of TBARS (Table 1). The difference can be explained by the additional antioxidative compounds delivered with acid whey and apple vinegar. Cysteine is the abundant amino acid of acid whey characterized by antioxidant properties that would have inhibited PUFA oxidation [82]. In vinegar, the antioxidant activity is related to the phenolic content. The acidic environment stabilizes vinegar polyphenols, increasing their antioxidant activity [15]. This is concordant with the results of Karwowska and Kononiuk's salted beef fermented sausages with or without the addition of acid whey. The content of PUFA in beef sausages with acid whey after 21 days of ripening was higher in comparison with salted samples. The antioxidative effect was enhanced by the acid whey protection of haem iron loss during meat ripening [15]. The n6/n3 ratio in the studied beef hams ranged from 1.84 (A variant after production) to 3.00 (AW variant after 8 months storage). The n-3 and n-6 fatty acids are crucial fatty acids for the human diet. Many studies have shown that the consumption of food containing n-3 and n-6 fatty acids is associated with a decreased risk of cardiovascular disease, insulin resistance, lipid profile, and obesity [83]. Research indicates that the balance between n-6 to n-3 polyunsaturated fatty acids (PUFA) in the diet is crucial in preventing atherosclerosis and metabolic disorders [84]. The optimal ratio in the human diet of n-6/n-3 PUFA is considered to be 5:1 [85]. The A ham variants, after 8 months of storage, had a ratio of 4.56, which was higher than C and AW treatments.

3.4. Volatile Compounds

The volatile compounds contribute to the unique sensory characteristics of fermented meat products [86]. During meat manufacturing and storage, muscle, endogenous, and microbial enzymes are responsible for the degradation of branched-chain amino acids, which, along with lipid oxidation and Maillard reaction, lead to the formation of various volatile compounds [7,87]. Some physical conditions, as well as the addition of preservation compounds, influence the biochemical processes and alter the composition of the volatile compounds and, as a result, the flavor generation of finished food products. Therefore, the meat product aroma characteristic is related to the composition and concentration of active volatile compounds exceeding the olfactory threshold [13]. A total of 60 volatile compounds were identified in the raw fermented beef hams. The identified volatile compounds belonged to many chemical groups: alcohols (11), aldehydes (11), carboxylic acids

(2), esters (15), (1), hydrocarbons (10), ketones (5), pyrazines (4), and others (2). Detailed results of the analysis of volatile compounds are shown in Table 4. The statistical analysis showed significant differences ($p < 0.05$) in the relative content of many individual volatiles depending on the treatment and storage time (Table 4).

Alcohols were one of the most abundant groups. Alcohols are associated with a characteristic fatty odor and are considered to be a product of a transamination and decarboxylation of branched-chain amino acid, as well as of the secondary oxidation of polyunsaturated fatty acids [11,13]. The storage of hams resulted in an increase in the alcohol content in the C samples and a decrease in the acid whey treatment. The ethanol content after production was the highest in the C treatment and decreased during storage in all variants of hams. In general, alcohols do not have a large impact on the aroma of dry fermented meat products due to their high odor threshold, although some of them have a low odor threshold and can potentially have a significant impact on the smell of these products. These include 1-octen-3-ol, associated with the mushroom/fungus-like aroma [88,89]. In the study of Marušić et al. (2014), 1-octen-3-ol was the most abundant alcohol in Istrian dry-cured ham meat [70]. In the studied hams, 1- octen-3-ol was present in low amounts (0.17–1.915%). After storage, the concentration of this alcohol increased in AW samples and was higher than in control hams. Another alcohol, 1-butanol, 3-methyl, which, at an appropriate concentration, can be responsible for a pleasant woody, acorn, and green odor [90] after production was present in the highest content in the ham treated with acid whey. Storage resulted in a decrease in this alcohol in the AW samples, while, in C variants, the content increased (Table 4). The stored A hams had a lower content of 2,3-butanediol S compared to C and AW variants. As a result of storage, the content of 2,3-butanediol S in the tested samples increased. 2,3-butanediol can be attributed to the metabolism of lactate and pyruvate catalyzed by LAB [91].

The second major group of volatile compounds in studied hams were aldehydes (11 aldehydes were detected in studied hams). The presence of aldehydes is associated with oxidative reactions of unsaturated fatty acids and proteolytic activity [92–94]. Aldehydes, due to their low odor threshold values, can contribute to the development of meat products' aroma [95]. Aliphatic saturated aldehydes are good indicators of oxidation occurring in raw-ripened hams. The total of all aldehydes after production in AW hams was lower than in the case of C and A treatment. During storage, in C hams, there was a decrease in the total content of aldehydes, and their content was lower than in the A and AW variants. In hams treated with acid whey, an increase in total aldehydes was observed during storage. This could be the result of an increase in the content of lipid-oxidation-derived hexanal in hams treated with apple vinegar and acid whey during storage, and an increase in the content of benzaldehyde in the case of the acid whey variant [96]. Benzaldehyde is a product of the amino acid catabolism oxidation of α-linolenic acid and the Maillard reaction of phenylalanine [97,98].

Hexanal is considered as a good indicator of the degree of oxidation and is associated with the aftertaste derived from the peroxidation of n-6 acids [99]. The presence of hexanal is attributed to the typical green-grassy fatty odor of dry-cured ham [90,100]. During storage, the increase in hexanal in hams treated with apple vinegar and acid whey may indicate active oxidative processes, which can be confirmed by a higher TBARS index observed in these variants of studied hams after 8 months of storage (Table 1).

Table 4. Volatile compounds in raw fermented beef hams at 0 and 8 months of storage and their relative abundance (means ± SD).

Volatile Compound	Rt	LRI	C		A		C vs. A		AW		C vs. AW		A vs. W	
			0 Months	8 Months	0 Months	8 Months	0 m	8 m	0 Months	8 Months	0 m	8 m	0 m	8 m
Alcohols														
Ethanol	1.663	549.7	9.97 ± 1.70 ^{b}	4.82 ± 0.13 ^{a}	4.83 ± 0.55	4.57 ± 0.06	*		9.22 ± 0.83 ^{b}	4.55 ± 0.08 ^{a}		*	*	
1-Butanol, 3-methyl	4.332	726.1	2.80 ± 0.55 ^{a}	4.69 ± 0.15 ^{b}	3.82 ± 0.19	3.44 ± 0.65	*	*	6.95 ± 0.92 ^{b}	1.93 ± 0.02 ^{a}	*	*	*	*
1-Butanol, 2-methyl	4.412	728.5	1.78 ± 0.34 ^{a}	3.38 ± 0.11 ^{b}	1.69 ± 0.29 ^{a}	2.59 ± 0.30 ^{b}		*	4.11 ± 0.28 ^{b}	1.77 ± 0.04 ^{a}	*	*	*	*
1-Pentanol	5.411	759.8	0.67 ± 0.08 ^{a}	1.24 ± 0.07 ^{b}	0.62 ± 0.02	0.49 ± 0.06		*	0.70 ± 0.09 ^{b}	0.22 ± 0.04 ^{a}		*		*
2,3-Butanediol, S	6.361	790.7	4.67 ± 0.97 ^{a}	8.97 ± 0.45 ^{b}	7.17 ± 0.90 ^{a}	7.38 ± 0.09 ^{b}	*	*	5.33 ± 0.16 ^{a}	9.62 ± 0.28 ^{b}			*	*
2,3-Butanediol, R	6.768	801.6	nd	nd	nd	nd			nd	nd				
1-Hexanol	11.409	871	1.13 ± 0.39 ^{a}	2.55 ± 0.07 ^{b}	1.15 ± 0.12	1.14 ± 0.03		*	0.26 ± 0.02 ^{a}	0.46 ± 0.03 ^{b}	*	*	*	*
1-Octen-3-ol	18.07	982.5	0.50 ± 0.01	0.49 ± 0.00	1.15 ± 0.33 ^{b}	0.87 ± 0.06 ^{a}	*	*	0.17 ± 0.02 ^{a}	0.96 ± 0.02 ^{b}	*	*	*	
1-Hexanol, 2-ethyl	20.6	1033.2	0.11 ± 0.03	0.09 ± 0.01	0.59 ± 0.24 ^{b}	0.15 ± 0.01 ^{a}	*		nd	0.23 ± 0.01	*	*	*	
Phenylethyl alcohol	24.132	1111.6	0.61 ± 0.19 ^{a}	2.06 ± 0.11 ^{b}	0.87 ± 0.30 ^{b}	0.28 ± 0.06 ^{a}		*	nd	0.31 ± 0.02	*	*	*	
Benzeneethanol	24.43	1117	0.12 ± 0.01	0.10 ± 0.03	0.27 ± 0.03	0.21 ± 0.07	*		nd	0.20 ± 0.07	*		*	
Total			22.37 ± 4.27 ^{a}	28.39 ± 1.12 ^{b}	22.18 ± 2.96	21.13 ± 1.40		*	26.74 ± 2.32 ^{b}	20.25 ± 0.62 ^{a}	*	*	*	
Aldehydes														
2-Methylpropanal	2.04	577.4	0.57 ± 0.02 ^{a}	1.01 ± 0.02 ^{b}	0.97 ± 0.01 ^{b}	0.56 ± 0.02 ^{a}	*	*	3.29 ± 0.18 ^{b}	0.50 ± 0.04 ^{a}	*	*	*	
3-Methylbutanal	2.79	641.2	2.79 ± 0.18 ^{b}	2.14 ± 0.30 ^{a}	3.46 ± 0.21	nd		*	1.24 ± 0.07	nd	*	*	*	
2-Methylbutanal	2.911	651.2	4.19 ± 1.34 ^{b}	2.01 ± 0.09 ^{a}	3.06 ± 0.22 ^{b}	2.03 ± 0.04 ^{a}	*		4.61 ± 0.56 ^{b}	1.55 ± 0.03 ^{a}		*	*	*
Hexanal	6.617	798.5	11.45 ± 0.60 ^{b}	5.32 ± 0.47 ^{a}	9.38 ± 0.34	10.05 ± 0.53	*	*	1.22 ± 0.09 ^{a}	10.58 ± 0.22 ^{b}	*	*	*	
Heptanal	13.354	900.4	0.45 ± 0.11 ^{a}	0.96 ± 0.14 ^{b}	0.72 ± 0.02 ^{a}	3.17 ± 0.10 ^{b}	*	*	0.77 ± 0.07 ^{a}	1.51 ± 0.15 ^{b}	*	*		*
3-(Methylsulfanyl) propanal	13.569	904.3	0.08 ± 0.03 ^{a}	0.21 ± 0.01 ^{b}	0.30 ± 0.04	nd	*	*	0.12± 0.01	0.25 ± 0.02			*	*
Benzaldehyde	16.664	957.8	3.54 ± 0.28 ^{b}	1.67 ± 0.04 ^{a}	1.40 ± 0.15 ^{b}	0.61 ± 0.09 ^{a}	*	*	1.24 ± 0.09	1.58 ± 0.12	*			*
Octanal	19.167	1002	1.47 ± 0.08 ^{b}	1.15 ± 0.02 ^{a}	0.95 ± 0.12 ^{a}	1.85 ± 0.17 ^{b}	*	*	1.43 ± 0.05 ^{a}	1.96 ± 0.09 ^{b}		*	*	
Benzeneacetaldehyde	21.003	1042	0.93 ± 0.27 ^{b}	0.50 ± 0.10 ^{a}	0.41 ± 0.08 ^{b}	0.20 ± 0.04 ^{a}	*	*	0.72 ± 0.05 ^{b}	0.22 ± 0.01 ^{a}		*	*	
Nonanal	23.819	1103.8	0.84 ± 0.02	0.95 ± 0.01	1.26 ± 0.14 ^{a}	3.48 ± 0.02 ^{b}	*	*	2.82 ± 0.45	3.25 ± 0.10	*	*	*	
Decanal	27.827	1204.7	0.07 ± 0.02	0.04 ± 0.00	0.08 ± 0.01	0.09 ± 0.00			0.05 ± 0.02	0.08 ± 0.00				
Total			26.37 ± 2.96 ^{b}	15.96 ± 1.20 ^{a}	22.01 ± 1.35	22.03 ± 1.02	*	*	17.50 ± 1.64 ^{a}	21.47 ± 0.79 ^{b}	*	*	*	
Acids														
Acetic acid	2.738	637.6	14.92 ± 0.75	15.37 ± 2.34	21.09 ± 2.57	22.15 ± 1.10	*	*	29.92 ± 1.90 ^{b}	23.59 ± 0.51 ^{a}	*	*	*	
Butanoic acid, 3-methyl	11.115	866.8	0.23 ± 0.01 ^{a}	0.41 ± 0.02 ^{b}	0.14 ± 0.01 ^{a}	0.40 ± 0.00 ^{b}			nd	0.07 ± 0.00	*	*	*	*
Total			15.15 ± 0.76	15.78 ± 2.35	21.23 ± 2.58	22.55 ± 1.10	*	*	29.92 ± 1.90 ^{b}	23.66 ± 0.52 ^{a}	*	*	*	

Table 4. *Cont.*

Volatile Compound	Rt	LRI	C		A		C vs. A		AW		C vs. AW		A vs. W	
			0 Months	8 Months	0 Months	8 Months	0 m	8 m	0 Months	8 Months	0 m	8 m	0 m	8 m
Esters														
Ethyl acetate	2.376	607.7	8.93 ± 0.07	8.52 ± 0.66	10.01 ± 0.60	10.93 ± 0.66	*	*	7.38 ± 0.56 ^{a}	10.75 ± 0.05 ^{b}	*	*	*	
Ethyl 2- methylpropanoate	5.049	748.7	0.67 ± 0.01	0.81 ± 0.06	0.55 ± 0.04	0.64 ± 0.01		*	1.37 ± 0.07 ^{b}	0.51 ± 0.01 ^{a}	*	*	*	
Isobutyl acetate	5.569	765.2	0.41 ± 0.06 ^{a}	0.82 ± 0.02 ^{b}	0.51 ± 0.01	0.61 ± 0.01		*	0.66 ± 0.03	0.69 ± 0.01	*		*	
Butanoic acid, ethyl ester	6.775	801.7	3.31 ± 0.67 ^{b}	1.74 ± 0.27 ^{a}	2.32 ± 0.46 ^{b}	1.83 ± 0.10 ^{a}	*		2.81 ± 0.07 ^{b}	2.08 ± 0.02 ^{a}		*		
Lactic acid, ethyl ester	7.503	812.2	1.21 ± 0.56 ^{b}	0.96 ± 0.06 ^{a}	1.45 ± 0.09 ^{a}	1.91 ± 0.20 ^{b}		*	1.21 ± 0.09 ^{a}	2.38 ± 0.06 ^{b}		*		
Butanoic acid, 2-methyl-, ethyl ester	9.803	847.1	0.42 ± 0.19 ^{a}	1.07 ± 0.07 ^{b}	0.47 ± 0.02	0.41 ± 0.01		*	0.32 ± 0.03	0.26 ± 0.03		*	*	*
Butanoic acid, 3-methyl-, ethyl ester	10.082	851.4	1.27 ± 0.50 ^{a}	7.26 ± 0.69 ^{b}	1.73 ± 0.15 ^{b}	1.30 ± 0.06 ^{a}	*	*	0.56 ± 0.02	0.68 ± 0.02	*	*	*	*
1- Butanol, 3-methyl-, acetate	11.899	878.6	0.46 ± 0.13 ^{a}	2.22 ± 0.15 ^{b}	1.63 ± 0.25	1.53 ± 0.04	*	*	0.34 ± 0.02 ^{a}	1.04 ± 0.05 ^{b}		*	*	*
1-Butanol, 2-methyl-, acetate	12.042	880.7	0.18 ± 0.05 ^{a}	1.25 ± 0.05 ^{b}	0.64 ± 0.06	0.53 ± 0.02	*	*	0.13 ± 0.02 ^{a}	0.36 ± 0.02 ^{b}		*	*	*
γ-Butyrolactone	13.961	911.3	0.14 ± 0.06	0.26 ± 0.02	0.54 ± 0.09	0.49 ± 0.07	*	*	nd	0.24 ± 0.01	*		*	*
1-Butanol, 3-methyl-, propanoate	17.554	973.5	0.71 ± 0.09 ^{b}	0.30 ± 0.01 ^{a}	0.38 ± 0.02	0.37 ± 0.02	*		0.34 ± 0.03	0.23 ± 0.01	*			
Acetic acid, hexyl ester	19.812	1016	nd	0.12 ± 0.01	0.33 ± 0.12 ^{b}	0.13 ± 0.01 ^{a}	*		nd	0.08 ± 0.00			*	
Octanoic acid, ethyl ester	27.555	1197.5	0.18 ± 0.01	0.15 ± 0.01	0.10 ± 0.02	0.12 ± 0.00			0.09 ± 0.01	0.17 ± 0.01				
Acetic acid, 2-phenylethyl ester	29.693	1256.5	nd	0.05 ± 0.00	0.05 ± 0.02	0.02 ± 0.00	*		0.02 ± 0.01	0.02 ± 0.00				
Decanoic acid, ethyl ester	34.428	1395.3	nd	0.02 ± 0.00	nd	0.01 ± 0.00			nd	nd		*		
Total			17.89 ± 2.39 ^{a}	25.56 ± 2.09 ^{b}	20.72 ± 1.94	20.83 ± 1.22	*	*	15.22 ± 0.96 ^{a}	19.50 ± 0.31 ^{b}	*	*	*	
Hydrocarbons														
1-Heptene	3.296	682.4	0.54 ± 0.02	0.53 ± 0.03	0.50 ± 0.03	0.46 ± 0.01			1.47 ± 0.08 ^{b}	0.55 ± 0.03 ^{a}	*		*	
3-Octene (E)	3.443	695	2.79 ± 0.99 ^{b}	2.10 ± 0.14 ^{a}	2.04 ± 0.19	2.03 ± 0.16	*		3.37 ± 0.07 ^{b}	2.04 ± 0.12 ^{a}			*	
Heptane	5.192	753.4	0.62 ± 0.09	0.50 ± 0.01	0.96 ± 0.09 ^{b}	0.71 ± 0.02 ^{a}	*	*	1.43 ± 0.13 ^{b}	0.96 ± 0.02 ^{a}	*	*	*	*
Toluene	7.296	809.6	0.34 ± 0.02	0.25 ± 0.02	0.40 ± 0.07	0.39 ± 0.02		*	0.43 ± 0.02	0.37 ± 0.02				
Benzene, 1,4-dimethyl	10.862	862.9	nd	0.28 ± 0.05	0.41 ± 0.06	0.60 ± 0.08	*	*	0.14 ± 0.01 ^{a}	0.42 ± 0.04 ^{b}	*	*	*	
Styrene	12.385	885.9	nd	nd	0.19 ± 0.03	nd	*		nd	nd			*	
o-Xylene	12.479	887.4	nd	1.11 ± 0.04	0.12 ± 0.02	0.14 ± 0.02	*	*	nd	0.17 ± 0.00		*	*	
2,2,4,6,6-Pentamethylheptane	18.424	988.6	0.30 ± 0.06 ^{a}	1.49 ± 0.09 ^{b}	1.49 ± 0.21 ^{a}	2.98 ± 0.71 ^{b}	*	*	nd	3.56 ± 0.06	*	*	*	*
Tridecane	31.208	1298.6	nd	0.02 c 0.00	0.04 ± 0.00	0.03 ± 0.00	*		0.01 ± 0.00	0.05 ± 0.00	*			
Pentadecane	37.644	1498.1	nd	0.01 ± 0.00	0.14 ± 0.04	0.03 ± 0.00	*		0.05 ± 0.01	0.10 ± 0.01	*			
Total			4.60 ± 1.18 ^{a}	6.30 ± 0.39 ^{b}	6.30 ± 0.73 ^{a}	7.38 ± 1.03 ^{b}	*	*	6.91 ± 0.32 ^{a}	8.22 ± 0.29 ^{b}	*	*		*

Table 4. *Cont.*

Volatile Compound	Rt	LRI	C		A		C vs. A		AW		C vs. AW		A vs. W	
			0 Months	8 Months	0 Months	8 Months	0 m	8 m	0 Months	8 Months	0 m	8 m	0 m	8 m
Ketones														
2-Propanone (Acetone)	1.746	557.2	2.54 ± 0.09 ^{b}	2.08 ± 0.06 ^{a}	1.89 ± 0.05 ^{b}	1.26 ± 0.03 ^{a}	*	*	1.41 ± 0.03	1.40 ± 0.01	*	*	*	
2,3-Butanedione	2.165	591.4	0.48 ± 0.03	0.60 ± 0.04	0.43 ± 0.01 ^{a}	0.78 ± 0.03 ^{b}			nd	0.79 ± 0.06	*		*	
2-Butanone	2.236	596	5.69 ± 0.79 ^{b}	0.86 ± 0.04 ^{a}	1.14 ± 0.06 ^{a}	1.66 ± 0.08 ^{b}	*	*	nd	1.08 ± 0.06	*		*	*
3-Hydroxy-2-butanone (Acetoin)	3.714	706.3	3.71 ± 0.29 ^{b}	2.44 ± 0.40 ^{a}	2.17 ± 0.07 ^{b}	1.10 ± 0.10 ^{a}	*	*	1.83 ± 0.08 ^{a}	2.47 ± 0.08 ^{b}	*			*
2-Heptanone	12.679	890.2	0.12 ± 0.00 a	1.32 ± 0.08 ^{b}	0.28 ± 0.04	0.21 ± 0.01		*	0.02 ± 0.00 ^{a}	0.24 ± 0.01 ^{b}	*	*	*	
Total			12.53 ± 1.21 ^{b}	7.30 ± 0.63 ^{a}	5.91 ± 0.23 ^{b}	5.01 ± 0.25 ^{a}	*	*	3.25 ± 0.11 ^{a}	5.99 ± 0.22 ^{b}	*	*	*	*
Pyrazines														
2,6-Dimethylpyrazine	13.904	910	0.28 ± 0.07	nd	0.34 ± 0.02 ^{b}	0.14 ± 0.02 ^{a}		*	0.33 ± 0.03 ^{b}	0.15 ± 0.01 ^{a}		*		
2,3-Dimethylpyrazine	14.311	917	nd	0.34 ± 0.03	0.17 ± 0.01	0.20 ± 0.01	*	*	nd	0.13 ± 0.00		*	*	
2,3,5-Trimethylpyrazine	18.982	998.4	0.42 ± 0.17 ^{b}	0.09 ± 0.00 ^{a}	0.58 ± 0.04 ^{b}	0.26 ± 0.06 ^{a}		*	nd	0.15 ± 0.00	*		*	
2,3,5,6-Tetramethylpyrazine	22.956	1084.5	0.04 ± 0.02	0.07 ± 0.01	0.09 ± 0.01	0.02 ± 0.00		*	nd	0.06 ± 0.00	*		*	
Total			0.74 ± 0.27 ^{b}	0.50 ± 0.04 ^{a}	1.18 ± 0.09 ^{b}	0.61 ± 0.09 ^{a}	*		0.33 ± 0.03 ^{a}	0.49 ± 0.02 ^{b}	*		*	
Others														
1, 8-Cineol	20.358	1027.9	0.03 ± 0.00	0.05 ± 0.01	0.12 ± 0.02	0.08 ± 0.00	*		nd	0.07 ± 0.00	*		*	
2-Pentylfuran	18.575	991.2	0.31 ± 0.04 ^{b}	0.17 ± 0.01 ^{a}	0.36 ± 0.03	0.39 ± 0.05		*	0.13 ± 0.00 ^{a}	0.36 ± 0.02 ^{b}		*	*	

Sample: Rt—retention time; LRI—linear retention index; method of identification of volatiles: mass spectra + LRI; AW—1.5% salt and 5% acid whey, A—1.5% salt and 5% apple vinegar, C—1.5% salt; n.d.—not detected. Values with different superscripts differ significantly ($p < 0.05$) regarding the storage time; values with asterisks differ significantly ($p < 0.05$) from the control sample regarding treatment; n = 3.

The short-branched-chain aldehydes 2-metylbutanal and 3-metylbutanal, due to a low odor threshold, are considered to be responsible for giving a characteristic malt, cocoa, and almond odor [101]. 2-metylbutanal and 3-metylbutanal can be formed in Strecker's degradation reactions from amino acids such as valine, leucine, and isoleucine, or as a result of the action of microorganisms in meat products [7,102]. During storage, there was a decrease in the content of these aldehydes in all variants of the studied hams.

The content of total carboxylic acids was the highest in the case of hams treated with acid whey after production. Stored samples of ham preserved with salt (C) had the lowest total of all carboxylic acids compared to stored hams treated with vinegar or acid whey. The most abounded was acetic acid. After production and after storage, its content in the hams preserved with salt (C) was lower than in the A and AW hams. This could promote the growth of acidophilic microorganisms, including LAB. An acidic environment can contribute to the microbiological safety and stability of the meat product. The meat sourness of the final product, at the appropriate level, may be appreciated by consumers preferring traditionally fermented products [13].

Esters play an important role in the formation of sensory characteristics of raw-ripened hams. They strongly affect the smell of hams, which is typical of ripened meat products. Esters are formed due to the bacterial esterification of acids and alcohols [103]. They have a low odor threshold and have a large share in developing the flavors of dry-cured products [104]. The acid-whey-treated hams had a lower total content of all esters after production in comparison with the C and A samples. As a result of storage, an increase in the content of total esters in the tested samples was observed. C, A, and AW hams did not differ in terms of the content of butanoic acid, ethyl ester. The content of this compound in all treatments of the stored samples was reduced, but the highest content was recorded in AW hams. The content of butanoic acid, 3-methyl-, and ethyl ester, responsible for fruity notes [90], did not change in the C and AW variants of hams during storage.

Hydrocarbons belonging to volatile compounds are derived from the reaction of lipid oxidation. They probably had no significant contribution to the final aroma [103]. The total amount of hydrocarbons in the studied hams did not differ after production and increased during storage. The total content of hydrocarbons in hams after the storage period was higher in the AW than in the C treatment hams.

Ketones have low threshold values and can contribute to the flavor of fermented meat products. The control hams (C) had a higher total content of ketones compared to the studied treatment methods. A similar difference occurred after the storage period, even though there was an increase in the total ketone content in A and AW hams.

Pyrazines are formed in Maillard reactions and can be responsible for pleasant aromas: nutty roast, earthy, and potato-like [13]. The abundance of all pyrazines was higher in the A hams than in the AW variant. After production, A ham samples had a higher total content of pyrazines than C and AW samples. According to Chen et al. (2023), the addition of exogenous amino acids can promote the formation of pyrazines [105]. Vinegar and acid whey contain pyrazines. The most abundant in the A variant was 2,3,5-trimethylpyrazine, which could be responsible for the roasted flavor [106]. As a result of storage, the total content of pyrazines was reduced.

3.5. Sensory Evaluation

Figure 1 ABC presents three kinds of fermented beef samples and the changes in sensory profile after 8 months of storage. The analysis of the data obtained in the QDP method regarding control sample C (Figure 1A) shows that the tested material during 8 months of storage at 4 °C changed significantly in terms of the sharp, storage, and other odor attributes, as well as in dried, sour, and other flavor notes. Moreover, a decrease in

juiciness and, finally, overall sensory quality was observed ($p < 0.05$). The same tendency was observed in the case of storage samples with apple vinegar addition (A) and with acid whey addition (AW) (Figure 1B,C). The period of 8 months of storage in low temperatures resulted in significant changes in sensory profile to a similar extent to compared to the control sample. The overall quality of control hams (C) and hams with the apple vinegar and acid whey was at a satisfactory level after production (over 6 c.u.) and after 8 months of storage (over 5 c.u.). Overall quality was decreased in all samples after storage, which could be caused by the increase in the intensity of the other odors, other flavors, and storage odors, as well as the decrease in the intensity of juiciness. Additionally, a significant increase in sour flavor notes was demonstrated.

The PCA analysis (Figure 2) showed that the first two principal components accounted for 71.68% of the total variability of all the sensory attributes of the tested samples. The PCA analysis showed that the overall quality of the ham samples was strongly and positively correlated with the smoked odor, tone of color, and smoked flavor, as well as juiciness (Figure 2).

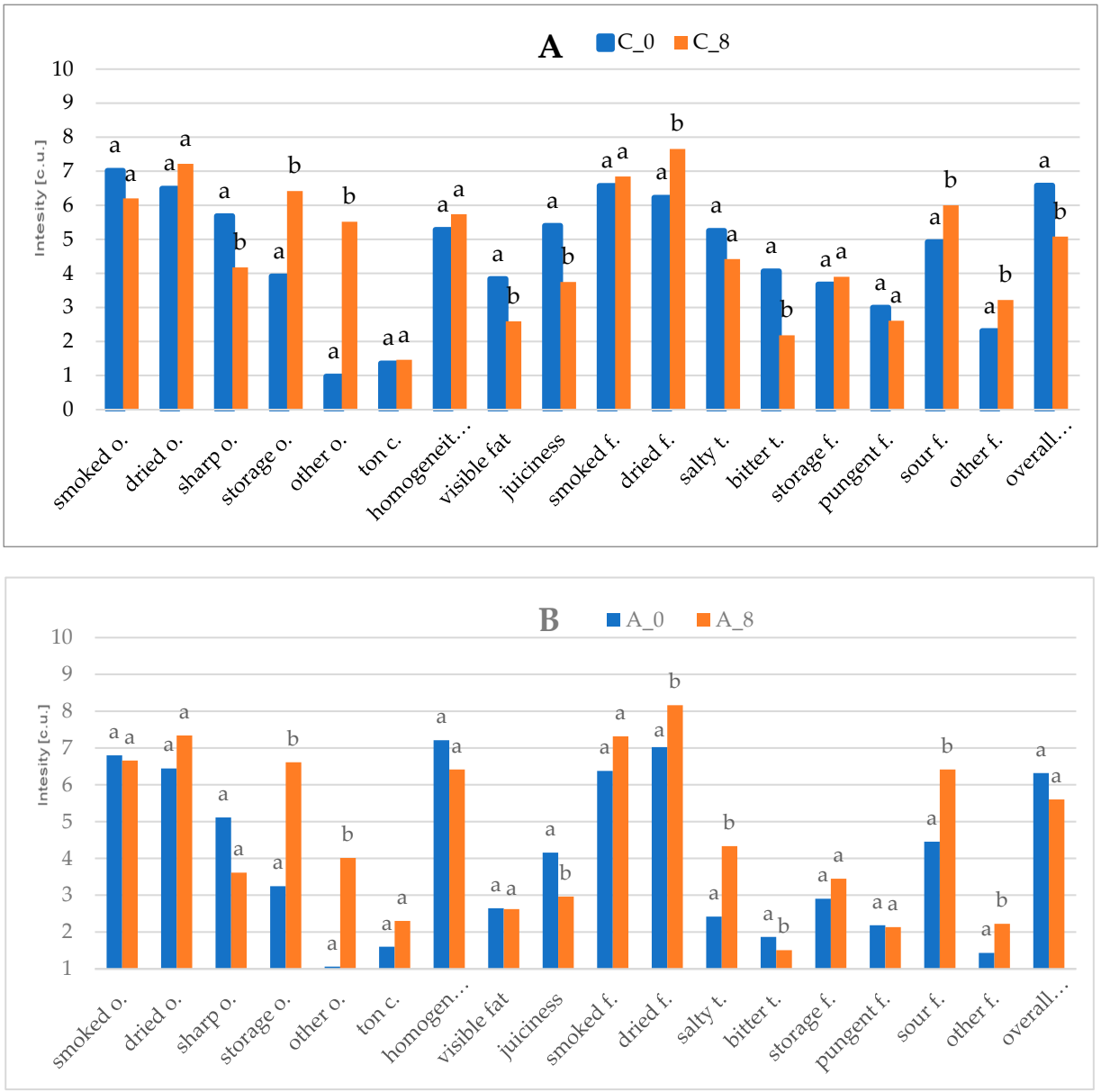

Figure 1. *Cont.*

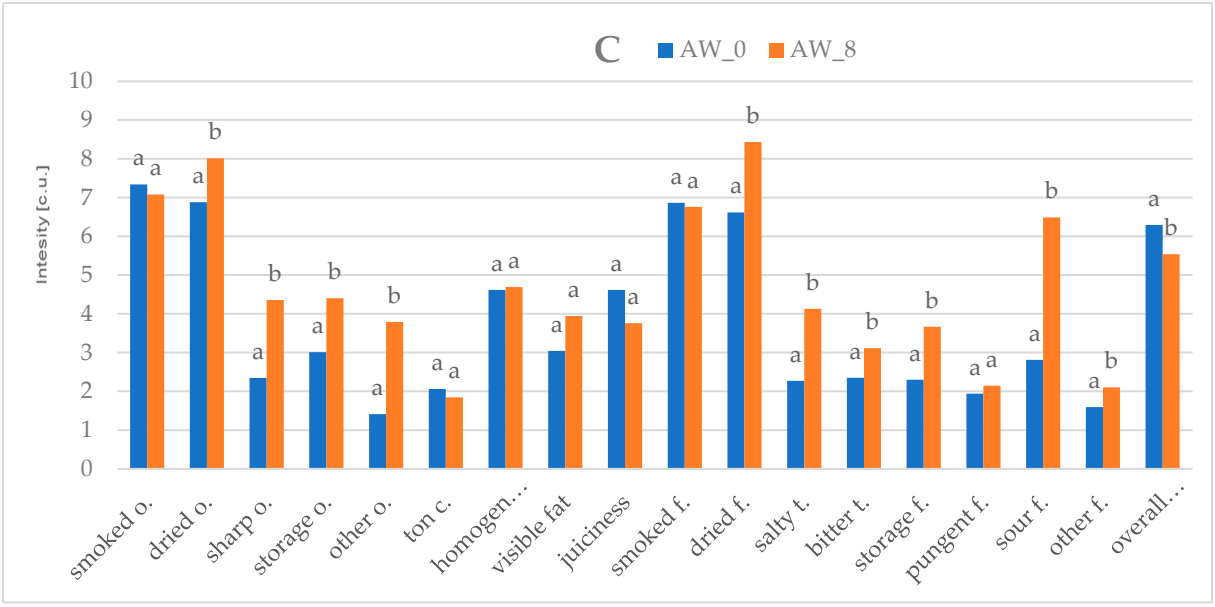

Figure 1. (**A**–**C**) Sensory attributes of fermented beef hams. Sample: control with salt: C_0—after production, C_8—after 8 months of storage; apple vinegar: A_0—after production, A_8—after 8 months of storage; acid whey: AW_0—after production, AW_8 —after 8 months of storage; o.—odor, f.—flavor; t.—taste; a, b—means statistically significantly for chosen sensory attribute ($p < 0.05$).

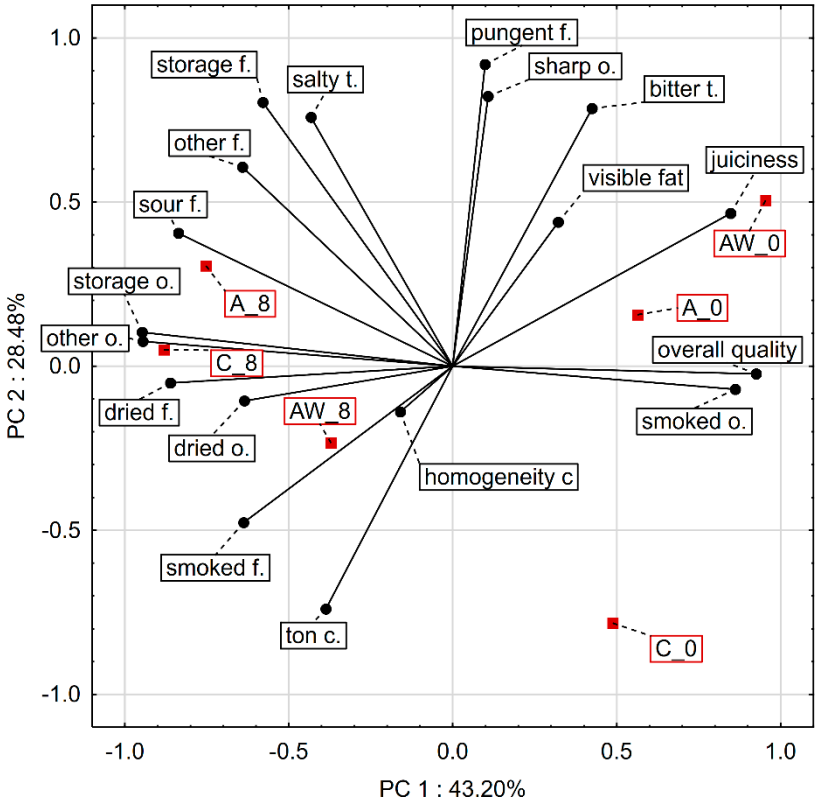

Figure 2. The PCA analysis. Sample: control with salt: C_0—after production, C_8—after 8 months of storage; apple vinegar: A_0—after production, A_8—after 8 months of storage; acid whey: AW_0 —after production, AW_8—after 8 months of storage; o.—odor, f.—flavor; t.—taste.

The storage at 4 °C for 8 months in the PCA plot indicates that the sensory quality of the samples changed significantly, as evidenced by the position of the samples on the opposite side of the overall sensory quality vector. Furthermore, the stored samples AW_8,

C_8, and A_8 are located near the vectors called storage odor, other odor, and dried odor and flavor. The position is related to the lower overall quality of the stored material.

In terms of the development of innovative meat products with reduced nitrates/nitrites and the addition of natural substances like organic acids, consumers expressed concerns related to taste, perceived healthiness, and shelf life of the final products. High values of the TBRAS index in the studied hams indicate the limited ability of acid whey and apple vinegar in the inhibition of lipid oxidation, which had a possible impact on sensory notes. In general, in the studies, consumers claimed to be willing to pay slightly more for innovatively fermented meats that promise to be healthier [107]. However, storage time decreased the overall sensory quality of the fermented products. The increase in negative notes like storage and dried notes influenced the quality. It is known from the literature that even a slight increase in the intensity of negative attributes, mainly flavor, and especially odor, is related to a severe decrease in the overall sensory quality of food products. These relations are not linear [108].

4. Conclusions

The raw fermented beef hams treated with acid whey or apple vinegar were characterized by moderate sensory quality after production and during storage. During storage, in the studied ham variants, an increase in the intensity of storage, other odors, and the sour dried flavor and salty taste were noted. Volatile compounds are formed in the process of the fermentation and storage of beef hams. It has been shown that the treatment with acid whey or apple vinegar modifies the formation and stability of volatile compounds, but does not affect the overall quality of hams.

Pathogenic bacteria were not detected in all the variants tested, confirming the safety of the final product. The number of lactic acid bacteria in raw fermented beef hams with acid whey and apple vinegar addition was high after 3 months of storage (approximately 7 log CFU/g), and decreased (approximately to 6 log CFU/g) after 8 months of storage. The presence of lactic acid bacteria at a high level during 3 months of storage has a preserving effect and guarantees the safety of the product, and may also have a positive impact on the health of the consumer. On the basis of the analyzed parameters (pH, TBARS, ORP, volatile compounds), the LAB viability, microbiological safety, and sensory assessment of both variants of natural beef hams (A and AW) are equally valuable. However, it should be emphasized that acid whey is a waste product of the dairy industry, and its application is ecologically and economically reasonable. The results indicate the possibility of reducing chemical preservatives through the use of acid whey or apple vinegar in the production of raw fermented beef hams. Organic fermented meat products enriched with naturally occurring preservatives and antioxidants are expected by a growing number of consumers. The development of new organic food products, along with chemical-preservative-free technologies, opens new perspectives for the food industry.

Acid whey and vinegar are rich in bioactive constituents and are suitable vectors for LAB delivery. However, for the implementation of acid whey or vinegar on a commercial scale into meat product processing, the possible drop in their quality should be considered.

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Institutional Review Board Statement: This study was carried out in accordance with the Declaration of Helsinki (World Medical Association 2013), and received approval from the Rector's Committee for the Ethics of Scientific Research Involving Humans at WULS-SGGW (Resolution No. 28/RKE/2023/U of 6 July 2023).

Data Availability Statement: The data presented in this study are available on request from the corresponding author.

Conflicts of Interest: The authors declare no conflict of interest.

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