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Investigating the Impact of Ultrasound, Microwave, and High-Pressure Processing of Milk on the Volatile Compounds and Sensory Properties of Cheddar Cheese

Masooma Munir ^{1,2,3}, Muhammad Nadeem ^{1,*}, Barkat Ali ³, Muhammad Sultan ^{4,*}, Rabia Kanwal ⁵, Huda Abdalrahman Al-Jumayi ⁶, Eman Hassan Ahmed Algarni ⁶, Maged B. Alnofeai ⁷ and Samy F. Mahmoud ⁸

- ¹ Institute of Food Science and Nutrition, University of Sargodha, Sargodha 40100, Pakistan; masoomamunir@parc.gov.pk
- ² School of Chemistry, The University of Melbourne, Parkville, VIC 3010, Australia
- ³ National Agricultural Research Centre, Food Science Research Institute, Islamabad 44000, Pakistan; bkfoodchem@yahoo.com
- ⁴ Department of Agricultural Engineering, Bahauddin Zakariya University, Bosan Road, Multan 60800, Pakistan
- ⁵ Post-Harvest Research Centre, Ayub Agriculture Research Institute, Jhang Road, Faisalabad 38850, Pakistan; rabiak_018@yahoo.com
- ⁶ Department of Food Science and Nutrition, College of Science, Taif University, P.O. Box 11099, Taif 21944, Saudi Arabia; huda.a@tu.edu.sa (H.A.A.-J.); eman1400@tu.edu.sa (E.H.A.A.)
- ⁷ Armed Forces Hospitals, Taif City 21944, Saudi Arabia; s.44180902@students.tu.edu.sa
- ⁸ Department of Biotechnology, College of Science, Taif University, P.O. Box 11099, Taif 21944, Saudi Arabia; s.farouk@tu.edu.sa
- Correspondence: nadeem.abdul@uos.edu.pk (M.N.); muhammadsultan@bzu.edu.pk (M.S.)

Abstract: Each cheese type has a unique flavor. A variety of compounds of various concentrations and different chemical classes contribute to this flavor. In the present study, the effect of processing techniques (ultrasonication (US), high pressure processing (HPP), microwave (MW)) on the flavors and sensory properties of milk and cheese was investigated. Samples of treated and untreated milk and cheese were analyzed for volatile compounds (including aldehydes, ketones, alcohols, fatty acids, and hydrocarbons). Significant variation was observed, and the results of sensory evaluation showed that cheese prepared from HPP and US-1 (21 J/g) secured higher sensory scores when compared to cheese prepared from US-2 (41 J/g), MW treatment, and the control. Variations in volatile profiles of milk and cheese were also evident according to the treatment process i.e., dodecanoic acid, octanoic acid, heptanoic acid, and nonanoic acid. Valeric acid was present only in US-1- (21 J/g) and US-2- (41 J/g) treated cheese, which is responsible for burnt and metallic flavors in milk and cheese. Butanoic acid, heptanal, heptanone, and butanediol were present in HPP, while ethyl butanoate, decanone, and 2,3-butanedione were present in microwave-treated milk. In a nutshell, the results of volatile compounds and sensory scores of cheeses are analogous, which shows that ultrasonication with high power output has some drawback because of the burnt and metallic flavor.

Keywords: cheddar cheese; HPP; microwave; sonication; volatiles; sensory characters

1. Introduction

Worldwide, various nutritionists recommend that milk and dairy products are included in the daily diet [1,2]. This is due to the presence of micro- and macronutrients and a plentiful concentration of minerals and vitamins [3,4]. Cheese is defined as the fresh or matured product prepared by converting fluid milk to a semisolid mass through the use of a coagulating agent such as rennet, acid, heat-plus-acid, or a combination of both. Cheese is a complex food matrix predominantly consisting of fat, proteins, moisture, and residual lactose that undergo biochemical reactions to form volatile compounds [5,6]. Aroma and flavor in cheese is developed due to the formation of volatile compounds and their interactions, and determine the consumer acceptability [7,8]. Volatile compounds are formed by



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Copyright: © 2022 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). the action of endogenous milk enzymes, rennet, starter bacteria, and secondary microbiota on milk fat, proteins, and carbohydrates during cheese maturation [9]. Therefore, milk quality, processing conditions, and the proteolytic activity of starter cultures determine the micro-environment of cheese for production of volatile compounds. Biochemical reactions like lipolysis, proteolysis, and the metabolism of residual lactose, lactate, citrate, free fatty acids (FFAs), and free amino acids (FAAs) by microorganisms present in starter cultures generate flavoring agents in cheese [5,6]. Free amino acids increase due to proteolysis, act as a substrate for catabolic reactions to produce different types of volatiles [10]. Similarly, lipolysis releases short-chain volatile free fatty acids [11]. Methyl ketones and secondary alcohols are low weight volatiles that are formed as a result of metabolism of lactate and citrate [12]. These flavor agents define the consumer selection and preference [13,14]. Thus, studying cheese flavor and other sensory and textural attributes is important for the evaluation of consumer acceptability [13].

Milk is heat-treated before cheese making to destroy pathogenic microbes. However, this also leads to thermal degradation of heat-sensitive compounds in the milk, including volatile compounds and loss of nutritional and sensory properties of the milk and resultant cheeses [15,16]. Therefore, to reduce unwanted effects of conventional thermal processing, alternative and novel dairy processing techniques such as ohmic heating [17,18], carbon dioxide technology [19], pulsed electric field [20], ultraviolet light [21], ultrasound [22–24], cold plasma [25], and pulsed-light [26] have been proposed. Of these technologies, ultrasound (US), microwave (MW), and high pressure (HP) processing of milk are becoming increasingly popular due to their efficiency and sustainability. The application of ultrasound in food processing has been used by many researchers for a long time. It involves application of high frequency (>18 kHz) sound waves to a liquid dairy stream. US causes pre-existing microbubbles in the liquid to oscillate and violently collapse in a process known as cavitation [26,27]. Bubble collapse leads to the formation of pockets of localized temperatures (ranging between 2000 and 5000 Kelvin), high pressure, and physical shearing [26]. US treatment has both positive and negative impacts on the characteristics of cheese milk. The rubbery aroma and off odors imparted by sonication are undesirable and could represent an impediment to what are potentially very useful features of the application of US in dairy product processing, such as the improvement of the rheological properties of yoghurt gels [28]. Physical shearing during US treatment breaks apart protein aggregates and fat droplets in cheese milk, improves their interactions, and accelerates milk coagulation. [29,30].

High-pressure (HP) treatment is a non-thermal method used for inactivation or reduction of pathogenic and spoiling micro-organisms in dairy matrices, which ensures safety during storage with minimal impact on sensory properties and nutritional value [31]. High-pressure technology could reduce microbiota in cheese thereby modifying the ripening indices [32]. Effective manipulation of high-pressure processing parameters has been utilized to accelerate cheese ripening or prevent over ripening [33] in order to achieve desirable flavor profiles within a specified maturation period. MW heating of milk occurs at a faster rate than in water for the same MW heating system, due to the presence of ionic components in the milk [34]. The main drawback of MW is the difficulty of distributing the temperature uniformly, resulting in hot and cold spots [35]. Earlier literature has investigated the effect of MW heating on milk composition, physical, and chemical properties [36–38].

The individual effects of US, MW, or HPP on the sensory properties of milk and milk products have been investigated, but a broader range of investigation is still lacking. Barring the comparative study of [39] that tested the effects of these three technologies on the antioxidant and anti-hypertensive properties of cheddar cheeses, their relative effects on the volatile compounds and the sensory attributes of cheeses have not been investigated. These techniques are gaining interest, due to their ability to alter the physico-chemical properties of milk components. Many food industries and researchers across the globe are adopting novel techniques for processing food products. Hence, it is of great importance

to compare the effect of these novel techniques on milk and cheese sensory characteristics after processing. Therefore, this study is aimed at investigating and comparing the effects of US, MW, and HPP pre-treatment of milk on volatile flavor generation, and to examine the sensory properties during cheddar cheese ripening. Insights gained during this study will help determine appropriate milk processing technologies that can lead to desirable flavor and sensory attributes in ripened cheddar cheeses.

2. Materials and Methods

2.1. Procurement of Raw Material

Pasteurized cow milk was purchased from a local market. The milk was standardized for fat content (3.4%) and protein content (3.3%), with the casein-to-fat ratio calculated to be 0.76:1 by the manufacturer. Starter culture *Lactococcus lactis* subsp. *lactis* and *Lc. lactis* subsp. *cremoris* (Cheese links, Vactoria, Australia) and rennet (200 IMCU/mL, Cheese links, Australia) were used to make cheddar cheese. All other chemicals were purchased from (Sigma-Aldrich, St. Louis, MO, USA).

2.2. Pre-Treatments

Green processing techniques (ultrasound: US, high pressure processing: HPP, and microwave: MW) were used to pre-treat the milk. Ultrasonication of milk was carried out at 80% amplitude with a probe sonicator (11 mm diameter horn, 20 kHz frequency, Sonifier 450; Branson, Danbury, CT, USA). Two sonication conditions (US-1 and US-2) were used for milk pre-treatment. During US-1, continuous sonication of the milk was carried out at a 15 mL/min flow rate (milk was passed-through twice) while US-2 sonication treatment was carried out at a 30 mL/min milk flow rate (double run). The calorimetric power delivered was 21 J/g and 41 J/g for US-1 and US-2, respectively. The bulk temperature of the milk streams during treatment was maintained below 40 °C using cooling water circulation. For high-pressure processing, milk was (5 bottles of 2 L) pressurized for 15 min at 400 MPa and 20 °C in a high-pressure vessel (Flow Pressure System QUINTUS® Food Press Type 35 L sterilization machine, Avure Technologies, Lincoln, NE, USA). Batches (1 L) of milk were microwave-treated with a specific energy of 86.5 J/g using a microwave oven (Samsung, Model No. ME6144W). During the treatment, the bulk temperature of milk did not exceed 40 °C. A total of 12 samples of milk were analyzed for different parameters.

2.3. Manufacturing of Cheddar Cheese

A total of 15 batches of Cheddar cheese were prepared in three independent replicated trials, with each batch made using 10 L of milk. Cheese was prepared following the procedure mentioned by Ong and Shah [40]. Before starting the cheese making, the milk pH was measured using a pH meter. A total of 15 samples of cheese were analyzed for different parameters.

2.4. Determination of Volatile Compounds in Milk and Cheese by GC-MS

Each cheese type made with differently pre-treated milks was analyzed for volatile compounds after 60 days of ripening [41]. Samples were taken from the cheese surface at a depth of 1 cm. Each cheese sample (7 g) was grated to a uniform grain size at 10–12 °C and 7 mL of 25% (w/v) NaH₂PO₄ (in milli-Q water) was added to the grated cheese sample. Milk samples were prepared similarly by mixing milk and 25% NaH₂PO in a 1:1 ratio on the basis of volume. Samples were equilibrated to 40 °C by placing in a water bath for 10 min while stirring the sample continuously. By using a septum, (DVB-CAR-PDMS; Supelco, Bellefonte, PA, USA) fiber was inserted into the vial. The fiber was positioned in the same way above the headspace of all the samples. For this, a solid phase microextraction (SPME) holder assembly was used. After the fiber was exposed to the headspace of the sample for 70 min at 50 °C, it was removed from the SPME assembly and from the sample vial. Then, volatile compounds were desorbed in a gas chromatograph. Between each sample run, the fiber was fully cleaned. Gas chromatographic analysis of the volatile compounds absorbed in

the SPME fiber was carried out on a GC (6890) equipped with a mass spectrometer detector (5973 MSD). Helium was used as the carrier gas and the total flow was kept 34.3 mL/min. The initial oven temperature was kept at 50 °C, post-temperature was 240 °C, and the maximum temperature was 325 °C. Other GC conditions are mentioned below.

2.4.1. Oven

The initial temperature of the oven was set at 50 °C, and the maximum at 325 °C. The initial time was for 2.00 min and equilibration time for 0.50 min. The post-temperature was set at 240 °C and post-time for 3.00 min. Run time was about 25.00 min, and ambient temperature set at 40 °C.

2.4.2. Front Inlet

The front inlet was set at split-less mode with an initial temperature of 250 $^{\circ}$ C (On) and pressure set at 2.00 psi (On), up to 19.37 psi (On). Helium was used as the carrier gas with a purge flow set at 30.0 mL/min and purge time for 2.00 min. Total flow was set at 34.3 mL/min with 30 mL/min gas flow rate for 4 min.

2.4.3. Column

A capillary column (model number: J&W 122-5532) of 30.0 m length, 250.00 um diameter, and 0.25 um film thickness was used. The column was operated at the maximum temperature of 325 °C with a 1.7 mL/min flow initially and 19.98 psi pressure, and average velocity of 34 cm/s.

2.4.4. Front Injector

The injection volume was 1.00 microliters and the syringe size was 10.0 microliters.

2.5. Sensory Evaluation of Cheddar Cheese

Sensory evaluation was conducted by a panel of 15 postgraduate students and 15 staff members of the institute using the methods reported by Ong and Shah [40] and Delgado-Martínez et al. [42]. All sessions for sensory evaluation were conducted at a temperature range of 20 °C to 25 °C in a sensory evaluation room equipped with white fluorescent light. Some panelists were already familiar with and some were briefed with basic sensory evaluation techniques for cheddar cheese. A piece of cheese (approximately 10 g) from each cheese was presented on a white plastic plate with a spoon. For better results, mineral water and a piece of bread was provided to each panelist between each sample. Samples were coded with random digits. Sensory evaluation for each cheese sample was conducted at same conditions at the start of ripening (0 day), 3, 6, and 9 months of ripening.

2.6. Statistical Analysis

All experiments were performed in triplicate and results obtained from our study were analyzed statistically by completely randomized design (CRD) and analysis of variance (ANOVA) technique [1]. The least significant difference (LSD) was used to separate means of significant treatments and storage time. Statistical analysis was done by using statistic 8.1 software (Analytical Software, Tallahassee, FL, USA). Results are presented as means \pm standard deviation and considered to be significantly different when *p* < 0.05 (at 95% confidence level).

3. Results and Discussion

3.1. Effect of Processing Techniques on Volatile Compounds of Milk

Volatile compounds in milk as a function of processing treatment (sonication, high pressure, microwave) are shown in Table 1. Volatile compounds were detected which were categorized into eight major chemical families: alcohols, acids, esters, aldehyde, ketones, hydrocarbons, furans, and benzene. All the volatile compounds showed significant variation in milk samples affected due to processing (Table 1).

| Volatile Compounds Alcohols | CAS No. | RT (Min) | Control (% Area) | HPP (% Area) | US (% Area) | MW (% Area) |
|--------------------------------|-------------|----------|---------------------------------|--------------------------------|--------------------------------|---------------------------------|
| Pentanol | 001320-98-5 | 1.5763 | $1.18\pm0.002~^{\rm a}$ | $1.15\pm0.003^{\text{ b}}$ | $1.15\pm0.002~^{\mathrm{b}}$ | 1.15 ± 0.004 $^{\rm b}$ |
| Hexanol | 000111-27-3 | 1.6246 | 0.81 ± 0.01 ^b | $0.76 \pm 0.03 \ { m b}$ | 1.02 ± 0.02 ^a | 0.79 ± 0.02 ^b |
| 2-methyl-3-Buten-2-Ol | 024509-88-4 | 1.7833 | ND | ND | 1.22 ± 0.05 ^a | ND |
| Octanol | 000589-98-0 | 1.804 | 2.97 ± 0.04 ^b | 2.89 ± 0.02 ^c | $3.144\pm0.06~^{\rm a}$ | ND |
| 2,6-Dimethyl-2-Heptanol | 013254-34-7 | 2.1486 | $0.558 \pm 0.001~^{a}$ | ND | ND | 0.609 ± 0.002 ^a |
| 3-Methyl-2-Buten-1-Ol | 000556-82-1 | 2.2522 | ND | 1.375 ± 0.01 $^{\rm a}$ | 1.387 ± 0.01 $^{\rm a}$ | ND |
| Isopentyl alcohol | 000123-51-3 | 3.6518 | 2.479 ± 0.01 $^{\rm a}$ | ND | ND | ND |
| 3-Methyl-1-Butanol | 000123-51-3 | 3.6794 | ND | ND | ND | $2.454\pm0.03~^{a}$ |
| Aldehydes | | | | | | |
| Heptanal | 000111-71-7 | 1.4936 | ND | 1.594 ± 0.004 ^b | 1.615 ± 0.002 ^a | ND |
| Ketones | | | | | | |
| Pentadecanone | 002345-28-0 | 1.6177 | 1.693 ± 0.02 ^a | 1.643 ± 0.01 ^{ab} | 1.670 ± 0.05 ^a | 1.596 ± 0.02 ^b |
| 2-Heptanone | 000110-43-0 | 1.6591 | 0.901 ± 0.003 ^d | 2.837 ± 0.03 ^b | 3.1718 ± 0.04 ^a | $1.289 \pm 0.001 \ ^{\rm c}$ |
| Octanone | 000111-13-7 | 1.7282 | 11.37 ± 0.11 $^{\rm c}$ | 11.69 ± 0.18 ^b | 12.88 ± 0.13 $^{\rm a}$ | 11.723 ± 0.12 ^b |
| 2-Butanonoe | 000078-93-3 | 2.0935 | 2.094 ± 0.005 ^b | 2.127 ± 0.006 ^b | 2.388 ± 0.02 a | 2.132 ± 0.02 ^b |
| Acetone | 000067-64-1 | 1.766 | 10.260 ± 0.034 c | 10.74 ± 0.01 ^b | 10.83 ± 0.05 ^a | $10.80\pm0.07~^{ m ab}$ |
| 2,3-pentadione | 000600-14-6 | 2.0452 | 1.1753 ± 0.002 ^a | ND | ND | ND |
| 3-octanone | 000106-68-3 | 2.0591 | ND | ND | $2.1237\pm a$ | ND |
| Hydrocarbons | | | | | | |
| Trichloromethane | 000067-66-3 | 2.2107 | 0.631 ± 0.001 ^c | 0.748 ± 0.00 ^b | 0.828 ± 0.002 ^a | 0.759 ± 0.004 ^{ab} |
| 4-Methyl-Heptane | 000589-53-7 | 3.4175 | ND | ND | 0.5914 ± 0.00 ^a | ND |
| 3-Ethyl-Pentane | 000617-78-7 | 3.4244 | $0.484 \pm 0.001 \ ^{ m c}$ | 0.466 ± 0.00 ^c | 0.701 ± 0.001 ^a | 0.577 ± 0.003 ^b |
| 2,3,3-Trimethyl-Pentane | 000560-21-4 | 3.5139 | 1.507 ± 0.002 ^a | ND | ND | 1.3684 ± 0.003 ^b |
| Hexadecane | 000544-76-3 | 3.541 | ND | ND | ND | $1.390\pm$ ^a |
| Decamethyl-cyclopentasiloxane | 0541-02-06 | 9.5956 | 16.23 ± 0.05 ^c | 16.15 ± 0.06 ^c | 18.54 ± 0.05 ^b | $21.72\pm0.08~^{\rm a}$ |
| 1-Hexene | 000592-41-6 | 1.9695 | ND | $1.022 \pm 0.00 \ ^{ m b}$ | 1.562 ± 0.005 ^a | ND |
| 2-Methyl-1-pentene | 000763-29-1 | 2.0039 | $0.8512 \pm 0.00~^{\rm c}$ | 1.053 ± 0.003 ^b | 1.85 ± 0.003 a | 0.615 ± 0.004 ^d |
| 1,2,3,6,7,8-Hexahydro-Pyrene | 001732-13-4 | 4.4518 | ND | ND | $5.563 \pm 0.01~^{a}$ | ND |
| 2,4-Dimethyl-1-Heptene | 019549-87-2 | 4.7689 | ND | 0.884 ± 0.000 | 1.229 ± 0.000 ^a | ND |
| Furan | | | | | | |
| Tetrahydrofuran | 000109-99-9 | 2.2383 | 2.1603 ± 0.002 ^a | ND | ND | ND |
| Acids | | | | | | |
| Butanoic Acid | 000107-92-6 | 4.1759 | $1.731 \pm 0.02^{\text{ a}}$ | 1.718 ± 0.01 ^a | 0.685 ± 0.01 ^b | ND |
| Valeric Acid | 000105-43-1 | 7.2789 | ND | ND | 1.039 ± 0.002 ^a | ND |
| Heptanoic Acid | 000111-14-8 | 7.3201 | ND | ND | ND | 1.894 ± 0.001 |
| Hexanoic Acid | 000142-62-1 | 7.3133 | 3.14 ± 0.02 ^b | 3.21 ± 0.002 ^b | 3.416 ± 0.005 ^a | 2.74 ± 0.003 ° |
| Octanoic Acid | 0124-07-02 | 10.2783 | ND | ND | 0.153 ± 0.00 ^a | ND |
| Benzene | 001014 (0.4 | 11 100 | NID | NID | 0.400 + 0.00 3 | ND |
| Benzene | 001014-60-4 | 11.409 | ND | ND | 0.423 ± 0.00 ^a | ND |
| Esters | | 0.00/5 | n a L a cost b | | | 0.00 + 0.001 3 |
| Ally Butanoate | 002051-78-7 | 2.2865 | 2.0 ± 0.001 ° | ND | | 2.38 ± 0.001^{a} |
| Amyl Isobutyrate | 002445-72-9 | 3.5209 | ND | 0.907 ± 0.002^{-0} | 1.035 ± 0.004 | ND |
| Methyl-Pentanoate | 000624-24-8 | 7.3684 | 2.223 ± 0.001 ^b | 2.260 ± 0.001 | 3.868 ± 0.009^{a} | ND |

Table 1. Effect of processing techniques on volatile compounds of milk.

Note: Numbers with different letters refer to a significant difference at 95%, ND refers to not detected. Here, RT is retention time, US is ultrasound, HPP is high hydrostatic pressure, and MW is microwave.

3.1.1. Alcohols

Chemical reduction of methyl ketones and aldehydes or through lactic acid bacteria dehydrogenases results in formation of different alcohols [43]. All the volatile compounds of the alcohol family showed significant variation in milk affected by different processing techniques. The maximum value (1.17%) for pentanol (fruity) was noticed in the control sample followed by US- (1.153%), HPP- (1.152%), and MW- (1.14%) treated milk. 2-methyl-3-Buten-2-Ol was only present in sonicated milk, while 3-Methyl-2-Buten-1-Ol (herbal, earthy, oily notes) was present in sonicated and HPP-treated milk but was absent in the control (without any treatment). Isopentyl alcohol was only present in the control milk samples. Hexanol (green, fruity, sweet green flavor) showed significant percent area while demonstrating all processing techniques. The maximum percent area (1.01%) was observed in US-treated milk and then in the control (0.80%), MW (0.78%), and HPP (0.75%). The maximum percent area (3.14%) for Octanol was observed in the US-treated milk, control (2.97%), and HPP-treated milk (2.89%). However, it was absent in MW treatment.

3-methyle-2-butene-1-ol (sweet fruity green flavor) showed maximum percent area (1.38%) for the US-2-treated milk followed by in the HPP-treated milk (1.37%) and it was absent in the rest of the treatments.

3.1.2. Aldehydes

Auto-oxidation of unsaturated fatty acids in milk results in formation of straight chain aldehydes. Decomposition of hydroperoxides generate aldehydes in milk [44]. The maximum amount of heptanal (fruity odor) was noticed in the US-treated milk, while it was not detected in the microwave-processed and unprocessed milk samples which undergo a decarboxylation process in the milk fat. The maximum pentadecanone (buttery, cheesy, meaty) was observed in the control followed by US- and HPP- and MW-treated milk. 3-octanone (mashroom like, cheesy) was only present in US-treated milk while 2-heptanone, Butanine and Acetone were present in sonicated milk (US-1) in high concentrations when compared to other treated samples.

3.1.3. Ketones

Ketones are naturally present in milk but these have different origin. All the volatile compounds of the ketone family showed significant variation for the percent area in the milk samples as affected by various processing techniques. The maximum percent area (1.69%) for pentadecanone (buttery, cheesy, meaty) was observed in the control, followed by in the US- (1.67%), HPP- (1.64%) and MW-treated milk (1.59%). When compared to other processing techniques, the maximum percent area (2.12%) for 3-octanone (mushroom like, cheesy) was noticed in the US-treated milk, however it was absent in milk samples treated with all other processing techniques.

3.1.4. Hydrocarbons

The origin of hydrocarbons is not well-known, however, aliphatic hydrocarbons may be present in raw, ultra-pasteurized [34], pasteurized, and fermented milk. The maximum concentration (0.82%) of hydrocarbons for (sweet) was observed in US-treated milk followed by MW- and HPP-treated milk. 4-Methyl-Heptane and trichloromethane was observed in US-treated milk, Hexadecane was present in microwave-treated milk, 1-hexene was noticed in milk samples treated with sonication processing followed by HPP-treated milk.

3.1.5. Acids

All the volatile compounds of the acid family showed significant variation for the percent area in milk samples as affected by different processing techniques. The maximum concentration of butanoic acid (cheesy sweat odor) was observed in the control followed by HPP-treated milk samples. however, it was absent in MW-treated milk. Valeric acid, that gives a burnt flavor, showed its presence in US-processed milk and was absent in the rest of the milk samples treated with various processing techniques including the control. Heptanoic acid (goaty rancid, soapy, pungent, fatty) was observed only in MW-treated milk, Octanoic acid (burnt, rancid like) was observed in the milk with an US processing technique and was absent in other milk samples.

3.1.6. Esters

All the volatile compounds of the esters family also showed significant variation for the percent area in milk samples as affected by different processing techniques. The maximum percent area (2.37%) for allyl butanoate (sweet, pungent) was observed in the MW-processed milk and it was absent in rest of the treatments. The maximum percent area (1.03%) for amyl isobutyrate (fruity, buttery) was observed in the US-treated milk samples, and then in the HPP-processed (0.90%) samples and was absent in all other treatments.

3.1.7. Furan

All the volatile compounds of the furan family also showed significant variation for the percent area in milk samples as affected by different processing techniques. The maximum percent area (2.16%) for tetrahydrofuran was observed in the control milk and it was absent in rest of the treatments.

Previous authors such as Valero et al. [45], Chouliara et al. [46], and Monteiro et al. [24] have reported on the volatile profile of milk. All changes in volatile compounds of milk are dependent upon processing techniques and treatments. For example, sonication has been proposed by previous researchers. Some changes in the volatile profile of sonicated milk samples were observed, some new compounds appeared, while some were missing in sonicated milk samples. These changes might be due to physical and chemical reactions occurring during sonication. It was reported that pyrolysis happens when cavitation bubbles collapse and is the main pathway for volatile compound generation and degradation. Karatapanis et al. [47] attempted to correlate sensory attributes and the volatile profile, but were unsuccessful. They reported that poor sensory attributes of milk after sonication are related to a number of volatile compounds rather than the presence of specific compounds [46]. While comparing our volatile profile of untreated control milk samples with treated milk samples, it was observed that nine new compounds appeared in the sonicated sample, four compounds in high-pressurized milk, and four compounds in the microwave-treated milk sample, which were not present in the control sample. Guimarães et al. [48] also reported more changes in the sensory and volatile profile of dairy beverages treated by sonication when comparing with conventional heat treatment.

Sonication is well-known to produce free radicals, and additional lipids may induce redox reactions under radical oxidation, extreme pressure, and temperature as result of bubble collapse [49]. All the suggested reactions and mechanisms may result in changes in the sensory characteristics of milk. Some scientists have reported burnt, rubbery, and plastic aromas to be linked with sonication treatment [46,49,50]. These aromas were linked to lipid oxidation, but some compounds with a strong smell have also been reported [47,51]. Thus, lipid oxidation induced by power ultrasounds or sonication might produce some aldehydes in milk. This rubbery-burnt aroma in milk caused by the sonication technique has an impediment to the useful potential features of sonication in different food systems. In our study, heptane is not present in the control and microwave-treated milk but is present in the sonicated milk sample. Cleavage of side chains of amino acids is also a direct route for production of hydrocarbons (benzene, toluene). On the other hand, sonication has induced many positive changes, i.e., improvement in the rheology and texture of yoghurt gels and other functionalities of milk protein [28,52]. Initial work has documented how the sonicated samples contained some new compounds which were not present in untreated samples. Chouliara et al. [46] and others tried to associate GC results with the sensory panel. Panelists reported burnt, distant, and chemical aromas in the milk. In this study, treatment was carried out for a shorter duration, at low-input power and lower temperature compared to Reiner's study, but the off flavors and aromas were still evident. Accordingly, they were also not able to detect a responsible volatile compound for this burnt aroma and smell. If these rubbery-smelling volatile compounds are the result of lipid oxidation, then these should not be present in skim milk (containing less than 0.5% milk fat). On other hand, very small amounts of fat in skim milk can be more easily oxidized as it is in a less integral form than the entire globules seen in total milk [49]. At this point we can say that the heat of sonication is also a very important factor: it is essential to control the heat produced by sonication. The adiabatic heating effect and applied pressure can cause thermal degradation of lipids, proteins, and sugars, which contribute to the formation of volatile compounds. But this mechanism is not enough to prove formation of volatile compounds in milk, as temperature rise is not as much as in other treatments such as pasteurization, and it has been reported that volatile compounds generated by HPP are different when compared to pasteurized milk. Pereda et al. [53] reported that the mechanisms of volatile formation under high pressure is not known, but they proposed

that the membrane of milk fat globules is more affected at high pressure, which results in exposed fatty acids which are involved in the formation of volatile compounds. As observed in our study, the size of fat globules is reduced, making them more vulnerable to reaction with enzymes. Enzymes can get easy access to fat and the higher surface area can induce lipolysis in milk.

HPP-treated milk had the least number of alcoholic compounds detected. This trend with HPP was also reported by Pereda et al. [53] where high-pressure-treated milk had a lower number of volatile compounds and generated aldehydes. In our study, heptanal is present in the HPP and sonicated milk samples but it was absent in the control. It might be due to oxidation of the fatty acid during the processing treatment of HPP and sonication. Sonicated milk had more numbers of alcoholic compounds. The increase in aldehyde concentration under application of pressure was also reported by Vazquez-Landaverde et al. [44]. They suggested that oxygen becomes more soluble under high pressure, and it could increase hydroperoxide formation, which leads to formation of more aldehydes. Monteiro et al. [24] studied sonicated milk and its volatile profile. They observed that more degradation of volatile compounds may occur by heat as compared to sonicated milk. Sonication even preserves these compounds when compared to pasteurization. Many researchers such as Chouliara et al. [46], Karatapanis et al. [47], and Yao, [54] have acknowledged similar profiles of volatile compounds in sonicated milk to those we observed in our samples. Some variations are noticed and can be due to differences in milk sources, animal feed, region, and treatment conditions.

Valero et al. [45] reported on the volatile profile of microwave treated milk. They did not observe qualitative differences between microwave- and conventionally heated milk. Our finding also supports this result, as microwave-treated milk was not much different to the untreated milk sample. Sonicated and high-pressure-processed milk had differences when compared to the control, but we found some compounds in the microwave-treated milk which were not present in the control samples.

3.2. Effect of Processing Techniques on Volatile Compounds of Cheddar Cheese

The synthesis of volatile compounds in cheese occurs because of complex biochemical processes which take place by action of enzymes and microbes [6,14,54,55]. It is well-known that biochemical processes, i.e., proteolysis, lipolysis, hydrolysis, breakdown of lactose, citrate, and lactate can induce the generation of volatile compounds. Metabolic and catabolic processes of fatty acids, and free amino acids also contribute to aroma-generating volatile compounds in fermented dairy products [5,6]. In our study, almost 75 volatile compounds were detected under eight major categories. Volatile compounds were grouped into their chemical families: alcohols, aldehydes, acids, ketones, esters, alkanes, furans, and benzene.

3.2.1. Alcohols

In the alcohols family five compounds were detected. It can be seen from Table 2 that all cheddar cheese samples have different alcoholic compounds which shows that sonication, high pressure, and microwaving have an impact on cheese aroma in different ways. Some compounds are only present in the control cheese while some new compounds have been observed in treated-milk cheese samples. This shows that different biochemical processes occur after different treatments on milk and cheese. Juan et al. [56] reported the presence of 2,3-Butanediol in high-pressure-treated and control cheeses. The contents of 2-butanol decreased in cheeses treated at pressures higher than 400 MPa as reported by Juan et al. [56]. Ethanol is an important compound that contributes to cheese flavor but it is not present in cheese prepared from US-2 treatment even though it is present in all other treated cheese. The primary alcohol detected was ethanol (dry dust, sweet alcohol notes) which was present in cheese prepared from all treated milk samples except US-2, while the most common secondary alcohol identified was 2,3-butanediol (sweet, fruity fusel oil, and wine-like) that was present in all cheese samples. The maximum concentration was found in HPP-treated cheese. Presence of ethanol and 2,3-butanediol in cheese was also reported

by [14]. Ethanethiol is present only in US-2 and it has a pungent smell, Iso-menthol (minty fruity flavor) is present in both the sonicated cheese samples.

| Table 2. Volatile compounds of ch |
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| Category | Volatile Compounds | CAS | RT | Control | Us-2 | Us-1 | HPP | MW |
|-----------|-----------------------------------|-------------|--------|-------------------------------|---------------------------------|----------------------------------|-------------------------------|-------------------------------|
| | 2,3-Butanediol | 000513-85-9 | 1.3283 | 0.63 ± 0.00 ^d | 4.54 ± 0.006 ^b | $1.8\pm0.001~^{\rm c}$ | 5.58 ± 0.003 $^{\rm a}$ | $1.58\pm0.003~^{\rm c}$ |
| | 2-Octanol | 000123-96-6 | 1.5902 | ND | $1.45 \pm 0.00 \ ^{\mathrm{b}}$ | 2.56 ± 0.001 $^{\rm a}$ | 1.84 ± 0.002 ^b | ND |
| Alcohols | Ethanethiol | 1975-08-01 | 1.804 | ND | 1.29 ± 0.002 $^{\rm a}$ | ND | ND | ND |
| | Ethanol | 000064-17-5 | 1.3007 | 0.45 ± 0.00 ^d | ND | $3.62\pm0.002~^{a}$ | 2.95 ± 0.005 ^b | $2.03 \pm 0.004~^{\rm c}$ |
| | Melaleucol | 000000-00-0 | 6.541 | ND | 1.36 ± 0.002 ^b | 1.06 ± 0.003 c | 1.59 ± 0.003 a | 0.98 ± 0.001 ^d |
| | Hexanal | 000066-25-1 | | 0.58 ± 0.00 a | ND | 0.17 ± 0.00 | 0.215 ± 0.00 | 0.51 ± 0.00 |
| Aldehydes | Pentanal | 000110-62-3 | 2.79 | ND | 1.93 ± 0.00 a | 1.88 ± 0.001 ^b | ND | ND |
| | 2-Methyl Undecanal | 000110-41-8 | 2.8038 | ND | ND | ND | 1.82 ± 0.001 a | ND |
| | 2-Heptanone | 000110-43-0 | 1.304 | $0.64 \pm 0.002~^{ m c}$ | $0.85 \pm 0.002~^{\rm c}$ | ND | 4.64 ± 0.001 a | 3.06 ± 0.001 ^b |
| | 2-Decanone | 000693-54-9 | 1.5764 | ND | ND | 1.94 ± 0.002 ^b | 2.67 ± 0.003 ^a | $1.05 \pm 0.002~^{\rm c}$ |
| | 2-Octanone | 000111-13-7 | 1.7281 | ND | 7.1 ± 0.002 ^a | ND | 6.0 ± 0.002 ^b | ND |
| | 2-Pentadecanone | 002345-28-0 | 1.7558 | 2.25 ± 0.002 $^{\rm a}$ | ND | ND | ND | ND |
| Ketones | 2,3-Butanedione | 0431-03-08 | 2.0108 | ND | ND | ND | 2.56 ± 0.003 ^b | 1.28 ± 0.001 ^b |
| rectories | 2,3-Pentadione | 000600-14-6 | 2.066 | ND | 3.48 ± 0.04 $^{\mathrm{a}}$ | 3.09 ± 0.001 ^b | ND | ND |
| | 5-Methyl-3-Heptanone | 000541-85-5 | 2.0868 | 2.35 ± 0.002 ^b | ND | ND | 2.92 ± 0.003 $^{\rm a}$ | ND |
| | 3-Hydroxybutnone | 000513-86-0 | 2.9487 | 1.49 ± 0.001 ^d | $3.63 \pm 0.04~^{c}$ | 7.17 ± 0.01 ^b | ND | 16.01 ± 0.02 $^{\rm a}$ |
| | 2-pentanone | 000107-87-9 | 2.6934 | ND | 1.7 ± 0.001 $^{\rm a}$ | 0.89 ± 0.003 ^b | 0.88 ± 0.00 ^b | 0.41 ± 0.00 ^c |
| | Acetone | 000067-64-1 | 1.7075 | ND | 3.66 ± 0.002 ^b | $3.78 \pm 0.001~^{a}$ | $3.47 \pm 0.001 \ ^{\rm c}$ | ND |
| | n-Hexane | 000110-54-3 | 2.0522 | ND | 6.20 ± 0.001 ^a | ND | ND | ND |
| | Trichloromethane | 000067-66-3 | 2.2454 | 1.31 ± 0.001 $^{\rm a}$ | ND | ND | ND | ND |
| | Cyclotrisiloxane, hexamethyl- | 0541-05-09 | 4.3278 | 6.57 ± 0.06 $^{\rm b}$ | $6.1\pm0.002~^{d}$ | $6.2\pm$ ^c | $8.36\pm$ ^a | $3.59\pm$ ^e |
| | Octamethyl- Cyclotetrasiloxane | 000556-67-2 | 7.1341 | 8.99 ± 0.11 $^{\rm a}$ | 7.31 ± 0.13 $^{\rm c}$ | $8.12\pm0.08~^{b}$ | ND | $6.92\pm0.08~^{d}$ |
| Alkanes | Tricosane | 000638-67-5 | 7.3547 | ND | ND | ND | 2.0 ± 0.002 a | ND |
| | Eicosane | 000112-95-8 | 8.0856 | ND | 2.58 ± 0.00 a | $2.42\pm {}^{b}$ | ND | ND |
| | Octadecane | 000593-45-3 | 8.0924 | ND | ND | ND | 2.1 ± 0.002 a | ND |
| | 2,6,10,14-tetramethyl-Hexadecane | 000638-36-8 | 8.0925 | ND | $1.71\pm$ ^a | ND | $0.54 \pm {}^{b}$ | ND |
| | Nonadecane | 000629-92-5 | 8.4028 | ND | 0.68 ± 0.001 ^a | 0.66 ± 0.00 ^b | ND | 0.64 ± 0.00 ^c |
| | Hexamethyl- Cyclotrisiloxane | 0541-05-09 | 8.6373 | $0.63\pm0.00~^{d}$ | $5.12\pm0.005^{\ c}$ | $7.22\pm0.007^{\:b}$ | 8.37 ± 0.03 a | $5.26\pm0.02~^{c}$ |
| | Heptadecane | 000629-78-7 | 8.7613 | ND | 0.33 ± 0.00 a | ND | ND | ND |
| | Hexadecane | 000544-76-3 | 8.7614 | $0.37\pm {}^{a}$ | ND | ND | ND | ND |
| | Decamethyl-Cyclopentasiloxane | 0541-02-06 | 9.6027 | $1.32 \pm 0.00 \ ^{\rm e}$ | 17.84 ± 0.12 a | 11.94 ± 0.15 ^b | 10.82 ± 0.11 ^c | 9.47 ± 0.09 ^c |
| | Ethyl Acetate | 000141-78-6 | 2.1281 | ND | 6.74 ± 0.01 $^{\mathrm{a}}$ | 6.7 ± 0.001 ^b | ND | ND |
| Ectors | Amyl Acetate | 000628-63-7 | 2.2039 | ND | 6.55 ± 0.003 $^{\rm a}$ | ND | ND | ND |
| Esters | Ethyl Butanoate | 000105-54-4 | 2.983 | ND | ND | ND | $2.68\pm0.001~^{\rm a}$ | ND |
| | Pentyl Isobutanoate | 002445-72-9 | 3.7416 | ND | 0.14 ± 0.00 ^b | 0.57 ± 0.00 ^a | ND | ND |
| Furan | Tetrahydrofuran | 000109-99-9 | 2.2798 | 0.94 ± 0.0 $^{\mathrm{a}}$ | ND | ND | ND | ND |
| | Acetic Acid | 000064-19-7 | 3.3072 | 5.09 ± 0.001 ^b | ND | ND | 7.61 ± 0.01 ^a | ND |
| | Nonanoic Acid | 000112-05-0 | 4.2243 | ND | 4.89 ± 0.003 ^a | $3.98 \pm 0.005 \ ^{\mathrm{b}}$ | ND | ND |
| | Butanoic acid | 000107-92-6 | 4.2863 | 1.08 ± 0.00 e | 8.13 ± 0.08 a | 3.59 ± 0.03 c | 6.97 ± 0.008 ^b | 2.08 ± 0.006 ^d |
| Acids | Isovaleric Acid | 000503-74-2 | 4.4657 | 1.07 ± 0.00 ^b | ND | ND | ND | 0.47 ± 0.002 ^a |
| | 3-Methyl-Valereic Acid | 000105-43-1 | 4.5484 | 1.06 ± 0.003 $^{\rm a}$ | ND | ND | ND | ND |
| | Hexanoic acid | 000142-62-1 | 7.2857 | ND | 1.8 ± 0.001 ^b | ND | 2.55 ± 0.001 $^{\rm a}$ | ND |
| | Heptanoic acid | 000111-14-8 | 7.2858 | ND | 2.83 ± 0.002 a | 2.8 ± 0.001 ^b | ND | ND |
| | Dodecanoic acid | 0143-07-07 | 7.2996 | ND | $4.88\pm0.001~^{\rm a}$ | 2.12 ± 0.001 ^b | ND | ND |
| | Pentanoic acid | 000109-52-4 | 7.3824 | $2.12\pm$ ^a | ND | ND | ND | ND |
| Benzene | 1-methyl-3-1-methyethyl-Benzene | 000535-77-3 | 7.9753 | ND | 0.68 ± 0.00 ^a | 0.5 ± 0.00 ^b | ND . | ND |
| Alkenes | Toluene | 000108-88-3 | 3.6795 | ND | 1.9 ± 0.001 ^b | $1.43\pm0.002~^{\rm c}$ | 0.94 ± 0.002 ^d | $2.42\pm0.002~^{a}$ |

Note: Numbers with different letters refer to a significant difference at 95%, ND refers to not detected. Here, RT is retention time, US-1 is ultrasound 21 J/g, US-2 is ultrasound 41 J/g, HPP is high hydrostatic pressure, and MW is microwave.

3.2.2. Aldehydes

Aldehydes are minor compounds in this cheese as they contribute less in cheese flavor and aroma [57]. The catabolic process of free amino acids present in cheese produces alcohols, aldehydes, acids, and amines [14,55]. Transamination reaction of some aromatic and branched-chain amino acids (methionine, aspartic acid) convert these amino acids into α -keto acids and this reaction is catalyzed by amino acid aminotransferase. The resulting α -keto acids are then further degraded to aromatic, branched chain aldehydes, acyl-CoA, and hydroxy acids [10,54].

Volatile compounds of the aldehydes (three compounds) family showed significant variation as affected by different processing techniques. HPP-treated milk cheese has higher contents of hexanal when compared to other cheese samples. Hexanal is characterized by green, slightly fruity, lemon, and herbal notes. It is the most common aldehyde present in cheese [14]. Considering pentanal (fruity, nutty, fermented, bready), the maximum was observed in US-2-processed milk cheese samples. These results show that aldehyde contents have been affected by processing treatments. Chouliara et al. [46] reported the presence of hexanal and pentanal in pasteurized and sonicated milk samples.

3.2.3. Ketones

Oxidation of free fatty acids to β -ketoacids occur, which further convert into methyle ketones through decarboxylation with loss of one carbon [12]. Although ketones are usual contributors to the typical aroma of dairy products they are not abundant in the aroma profile of some cheeses [41]. 2-heptanone was observed in cheese prepared from HPPtreated milk and it was absent in ultra-sonicated cheese. 2-decanone and 2,3-butanedione was only noticed in cheese prepared from MW-treated milk and it was absent in the cheese samples prepared from milk treated with other processing techniques and the control. 2-Octanone (floral, green cheese-like smell) and 2-heptanone (spicy, musty flavor, blue cheese notes) were only observed in HPP-treated cheese. Considering 3-hydroxybutanone, this was noticed in the MW followed by the US-1, US-2, and control and it was absent in HPP. These variations in the aromatic profile of different cheddar cheeses depict the effect of processing on the volatiles of the cheese. Juan et al. [56] studied the impact of high pressure (400 MPa) on pasteurized ewes' milk cheese. They reported that high pressure can interfere with enzyme activity which is responsible for degradation of methyl ketones to alcohols, which results in a decreased level of alcohols and increased level of methyl ketones. On the other hand, high pressure can also help the generation of methyl ketones from free fatty acids. 2,3-butanedione was significantly higher in cheeses treated at 400 and 500 MPa than in the control cheese [57]. According to Curioni and Bosset [58], methyl ketones impart blue cheese notes, while other methyl ketones like 2-nonanone, 2-undecanone, and 2-decanone provide fruity, floral, and musty smells. Guimarães et al. [48] reported the presence of some ketones in US-treated whey beverages.

3.2.4. Hydrocarbons

All the volatile compounds of the alkene family showed significant variation for the percent area in cheese samples as affected by different processing techniques. n-Hexane, which has a fuel- or petroleum-like smell, was observed in those cheese samples which were fed under the US-2 processing technique, while these volatile compounds were absent in the rest of processing techniques including the control. Considering other volatile compounds of alkanes, the maximum percent area for sweet-tasting trichloro methane and Hexadecane were observed in the control and these compounds were absent in the cheese samples of the rest of processing techniques. Tricosane (waxy flavor) was noticed in the cheese samples which were prepared from milk subjected to the HPP processing technique while it was absent in the rest of the treatments including the control. Considering Eicosane (waxy odor), the maximum percent area was noticed in the US-2 followed by in the US-1, while it was absent in the rest of the processing treatments including the control. The maximum percent area for nonadecane was observed in the US-2 followed by the US-1. Heptadecane (bland flavor) showed the maximum percent area (0.33%) in the US-2 and it was absent in the rest of the cheese samples.

3.2.5. Alkenes

The maximum percentage for toluene was observed in the cheese samples prepared from milk subjected to MW treatment followed by the US-2, US-1, and HPP. It was absent in the control cheese samples. Toluene is associated with a nutty and rancid odor.

3.2.6. Esters

Esters play an important role in the aroma of cheese and they contribute a fruity flavor to cheese and other fermented products. Aldehydes have a low detection threshold when compared to esters, as esters are more volatile at ambient temperature [59]. Esters are involved indirectly in the metabolism of free fatty acids, alcoholysis, and the esterification process of free fatty acids leading to biosynthesis of esters. These reactions also mediated by microbial esterase [59,60]. Formation of esters from alcohols and carboxylic acids by action of esterase is called esterification, while formation of esters from alcohols and acylglycerols, or acyl-CoA by action of acyltransferase is called alcoholysis [60]. Some esters are characterized by their fruity, sweet, and floral notes and they have a low perception threshold. They have ability to minimize the bitterness and sharpness of cheese [58,60]. For ethyl butanoate (fruity, green apple, flowery odor) maximum concentration was observed in the MW. Pentyl Isobutanoate (fruity, apricot) showed its maximum concentration in the US-1 followed by the US-2 and it was absent in the rest of the processing techniques. Ethyl acetate and amyl acetate are present in sonicated cheese only while ethyl butanoate is present in HPP-treated cheese which is evidence that treated cheese samples have different aroma profiles when compared to untreated cheddar cheese samples. The overall aroma of this cheese is enhanced due to ester modifications, as they add fruity notes and have low insight verges [58].

3.2.7. Acids

Acids are another family of volatile compounds; short-chain fatty acids are formed by lipolysis but some acids are formed by lactose fermentation into lactic acid. Butanoic acid and ethanoic acid are formed by this fermentation process [61]. Lipid oxidation and deamination of amino acids also subsidize the overall acid pool [62]. In many ripened cheese varieties, the aroma of cheese given by acids has been demonstrated [11,32,61].

n-Butanoic acid is one of the most important short-chain free fatty acids responsible for the flavor in ewe milk cheeses and, like n-hexanoic acid, is characterized as having a strong and pungent cheese note [63]. n-pentanoic acid has been detected as a mild to strong savory cheese aroma [64]. Maximum percent area for 3-methyl-valeric acid and pentanoic acid (nutty, sour, sweaty, meaty) was noticed in the control and all these compounds were absent in the rest of the processing techniques. Maximum concentration for isovaleric acid (sour, sweet, rancid, cheesy, fruity, waxy) and dodecanoic acid (metallic, soapy) was highest in the US-2 followed by the US-1 and was absent in other treated cheese.

Marchesini et al. [51] reported that metallic flavor was due to dodecanoic acid and that burnt flavor resulted from octanoic acid. They found a significant correlation between volatile compounds and their specific aromas, and that these compounds affect sensory characteristics of the product [51]. HPP-processed cheese showed the maximum acetic acid (sour, pungent) followed by the control and it was absent in the rest of the processing techniques. Heptanoic acid (goaty rancid, soapy, pungent, fatty) and nonanoic acid (fatty, soapy, waxy, green) were present only in the US-1 and US-2 treated cheeses. Hexanoic acid (goaty, sharp, cheesy, sweaty) was detected in HPP and US-2 cheese only and the maximum area for butanoic acid (sharp, acidic, cheesy) was observed in the US-2 followed by the HPP and US-1 processing techniques respectively.

All changes in volatile compounds among treatment might be associated with proteolytic activity and maillard reactions occurring in the cheese [14,65]. Previous scientists have reported similar volatile profiles of cheddar-type cheese: [66–68] reported on the presence of alcohols, acids, esters, aldehyde, and ketones in surface-ripened cheeses and enzymemodified cheeses, respectively. It can be seen from the results that sonication treatment induced more changes in the flavor profile of the cheddar cheese. Guimarães et al. [48] also reported more changes in the sensory and volatile profile of dairy beverages induced by sonication when comparing with conventional heat treatment. José Delgado-Martínez et al. [42] reported that the volatile profile of raw ewe's milk cheese and its sensory characteristics was affected by high-pressure processing treatments. They observed a slight increase in the concentration of volatile compounds at 200 MPa pressure and a decrease in concentration at 600 MPa pressure.

3.3. Effect of Processing Techniques on Sensory Quality of Cheddar Cheese

Organoleptic and sensory evaluation is one of most important and useful parameters to assess quality of end product. In cheese and other fermented products, flavor and taste development is dependent upon milk quality, microbial activity, enzyme efficiency, and processing and ripening conditions. Complexity of the microbial population and chemical reactions contributes to formation of flavor and volatile compounds which impart characteristic flavor, taste, and aroma to cheese [14].

Significant variation ($p \le 0.05$) was observed for the sensory scores of cheddar cheese as affected by different processing techniques at various month's intervals during the process of cheese ripening. The maximum score for the flavor (7.70 ± 0.10) was noticed in the treatment where US-1 processing technique was applied and the sample was analyzed after 9 months of the ripening process. That was followed by the HPP treatment (7.67 ± 0. 21) in which the sample was analyzed at the same time interval. The minimum score for the flavor (5.00 ± 0.01) was noticed in those samples which were observed at the start of the ripening process in the control (Figure 1). The maximum score for the color (7.66 ± 0.15) was noticed in treatment where the HPP processing technique was applied, and the sample was analyzed after 9 months of the ripening process. That was followed by the US-1 (7.60 ± 0.28) in which the sample was analyzed at the same time interval. The minimum score for the color (4.86 ± 0.23) was noticed in those samples which were observed at the start of the ripening process in the MW-processed cheese.



Figure 1. Effect of processing on sensory scores of cheddar cheese. Error bars refer to standard deviation. Here, US-1 is ultrasound 21 J/g, US-2 is ultrasound 41 J/g, HPP is high hydrostatic pressure, and MW is microwave.

The maximum score for the taste (7.77 \pm 0.36) was noticed in the treatment where the HPP processing technique was applied, and sample was analyzed after 9 months of the ripening process. That was followed by the US-1 (7.67 \pm 0.12), in which the sample was analyzed at the same time interval. The minimum average score for the taste (5.07 \pm 0.21) was noticed in those samples which were observed at the start of the ripening process in MW-processed cheese. The maximum score for the texture (7.73 \pm 0.25) was noticed in the treatment where the US-1 processing technique was applied and the sample was analyzed

after 9 months of the ripening process. That was followed by the HPP (7.70 \pm 0.20), in which the sample was analyzed at the same time interval. The minimum average score for the texture (4.90 \pm 0.17) was noticed in those samples which were observed at the start of the ripening process. The maximum score for the overall acceptability (7.77 \pm 0.21) was noticed in the treatment where the US-1 processing technique was applied and the sample was analyzed after 9 months of the ripening process. That was followed by the HPP (7.73 \pm 0.14), in which the sample was analyzed at the same time interval.

Ripening had a significant effect on the sensory properties of different cheeses produced in this study. The maximum sensory score was filed for the cheese after 9 months of ripening, followed by 6 and 3 months. These results might be explained by the enhanced proteolysis during the ripening period as the products of proteolysis are the major contributors towards the sensory properties of cheese [42]. The maximum scores for color, flavor, texture, and overall acceptability by the judge's panel were observed for the HPP-treated-milk cheese. These results supported the notion that the release of certain volatile compounds and free amino acids might have developed the better attributes of the aroma and flavor. These results are in accordance with Voigt et al. [69], who reported that the initial whiteness (L-value) decreases because of the dissociation of casein micelle, but afterwards L-value increases, which might be the result of reassociation of destabilized casein to form larger aggregates, hence improving the color of the end product. Further, more soft, chewy, and gummy cheddar cheese is reported by Voigt et al. [70] and was made by pressurized milk. These results are also endorsed by Avila et al. [57], who reported that with pressure treatment of milk at less than 400 MPa, cheese with a buttery odor was produced. In the case of sonicated milk cheese, a higher score was observed for the US-1 treatment that was on par with the pressure treatment. The most efficient and effective treatment in this overall study, US-2, could not catch the attention of the sensory panel judges and received lower scores than the US-1. High-intensity sonication is thought to produce the free amino acids and volatile compounds with odd smells. However, in the case of the US-1, less power/intensity resulted in lesser volatile and free amino acids when compared to US-2, and might have attributed in better scores. In past studies several scientists have documented a burnt, rubbery, or plastic aroma in cheeses produced from sonicated milk [46,49,50]. Marchesini et al. [51] attributed this burnt odor towards the presence of elevated levels of octanoic acid. Beside the negative impact of the aroma, a uniform texture without any sedimentation and a positive mouth feel was documented by Jeličić et al. [71]. They further reported that there was no negative impact on color change during the whole process. There was no significant difference observed in the MW-processed milk cheese and unprocessed milk cheese. These results indicate that MW do not impose serious changes to proteins before or after ripening as the sensory attributes are basically related to the proteolytic products (volatile compounds and free amino acids) during the ripening period. These results agree with the findings of Ong and Shah, who reported that flavor and taste attributes in cheddar cheese are mainly due to the volatile compounds and free amino acids produced as a result of proteolysis. These results are also in accordance with those documented by Valero et al. [45], who reported that MW does not impart any difference in sensory attributes of cheeses when compared to the control and even the expert panelists were not able to differentiate between the MW-processed and conventionally pasteurized milk cheeses. While some contradictory results are also evidential in past studies such as the article by Clare et al. [72], where good sensory attributes in MW-pasteurized milk cheese was scored higher than conventionally pasteurized milk cheese. Similarly, Villamiel et al. [73] also reported on the improved sensory properties of milk when pasteurized by microwaves. These contradictions may prevail depending on duration and intensity of treatment.

4. Conclusions

In the dairy industry, conventional thermal processing has been widely used over recent decades. It is known that high temperatures can kill microbes but also lead to thermal degradation of thermal-sensitive compounds, i.e., volatile compounds. The physical and functional properties of milk proteins can be significantly altered with MW, HPP, and US technologies. In many cases these alterations can enhance processing and the properties of the final product. Volatile compounds in milk and cheese as a function of processing treatment (sonication, high pressure, microwave) were investigated in this study. Volatile profiles of milk and cheese and a score of sensory evaluation revealed that HPP- and US-1-treated cheese was liked most, while US-2-treated cheese had some volatile compounds which give a rubbery, burnt aroma and flavor. Therefore US-2 secured lower sensory scores while bioactivity of US-2 was highest when compared to other treatments. From the critical analysis of previous studies and the results of our studies, it can be concluded that the processing of milk systems using HPP, MW, and US technologies is still in the early stages of development, despite providing several positive attributes to the milk systems. The adaptation of these novel technologies by the dairy industry is a slow process and further research is needed for efficient implementation of these technologies in the dairy industry and other food industries.

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