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Comparative study of hot-air, freeze, and supercritical carbon dioxide drying on the physicochemical, microbiological, and sensory quality of shrimp (*Metapenaeus* spp.)☆

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ABSTRACT

This study evaluates the application of supercritical CO_2 drying on shrimp (*Metapenaeus* spp.) as a combined dehydration and decontamination strategy, comparing it to conventional hot-air drying (AD) and freeze-drying (FD). Two complementary storage assessments were performed over 4 months: microbiological stability under ambient storage conditions at 20 ± 1 °C, and physicochemical, structural, and volatile quality retention under refrigerated storage conditions at 4 ± 1 °C. All three treatments reached a comparable target water activity of approximately 0.55, achieved in 6.5 h (AD), 15 h ($scCO_2$), and 104 h (FD). Microbiologically, $scCO_2$ drying demonstrated the highest efficacy, maintaining natural spoilage flora at or near the detection limits throughout storage. A small-scale challenge test performed on $scCO_2$-dried samples achieved >4 Log CFU/g reductions for surrogate pathogens (*Listeria innocua* and *Enterococcus faecium*), confirming the antimicrobial potential of this technology. Moreover, physicochemical analyses of samples under refrigerated storage conditions at 4 ± 1 °C revealed that $scCO_2$ exhibited intermediate color preservation and rehydration capacity (rehydration ratio > 4 g/g), closer to FD than to AD, while maintaining a more stable volatile organic compound (VOC) profile. However, instrumental and sensory texture evaluations indicated that $scCO_2$-dried shrimp exhibited a firmer texture and a whitish appearance, both negatively perceived by consumers, resulting in consumer liking scores comparable to AD but lower than the highly porous FD samples. Overall, $scCO_2$ drying showed excellent microbiological safety under ambient storage conditions at 20 ± 1 °C and favorable physicochemical stability under refrigerated storage at 4 ± 1 °C, however further optimization is required to enhance its structural and sensory acceptance.

1. Introduction

Fish and seafood are highly nutritious commodities, providing easily digestible proteins, essential amino acids, omega-3 long-chain polyunsaturated fatty acids, vitamins (D and B12), and minerals (Khalili Tilami & Sampels, 2018; Lund, 2013; Monteiro et al., 2024).

Despite their remarkable nutritional profile, fish and seafood are highly perishable goods. Their rapid deterioration is primarily due to their unique biochemical composition, which makes them particularly susceptible to spoilage processes (Baptista et al., 2020; Patil & Khandekar, 2025). The high levels of free amino acids and unsaturated fatty acids, low amounts of connective tissue, and elevated enzymatic activity accelerate post-harvest changes such as protein degradation, lipid oxidation, and consequently the development of

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undesirable odors, flavors, and textures (Rabiepour et al., 2024; Ying et al., 2025). Consequently, the high perishability of fishery products necessitates the implementation of effective processing and preservation strategies to preserve their quality and safety throughout the supply chain (Kontominas et al., 2021).

While freezing is the most conventional method to preserve these commodities, it relies heavily on continuous cold chain logistics, which are highly energy-intensive and costly (Magnussen et al., 2008). In this context, drying represents a well-established and advantageous alternative to overcome the limitations of freezing. By reducing water activity and inhibiting microbial growth, drying not only extends the shelf life of seafood but also allows for storage at ambient temperatures, thereby significantly lowering energy requirements and transportation costs (Mujumdar, 2020). Hot air drying (AD) is the most commonly employed dehydration method in the food and chemical industries. However, the relatively high temperatures typically used in this process (60–90 °C) can adversely affect the microstructure of seafood products, leading to detrimental changes in color, texture, taste, aroma, and nutritional quality (Lin et al., 2025). Conversely, freeze-drying (FD) involves freezing the water content in the product, followed by sublimation of the ice directly into the vapor phase through controlled manipulation of pressure and temperature, yielding premium quality, highly porous products (Harguindeguy & Fissore, 2020). However, freeze-drying is hindered by high operational costs, high energy consumption, and long processing times (Pravallika et al., 2023). Furthermore, freeze-drying is not effective for microbial inactivation; on the contrary, it is commonly used as a method to preserve microorganisms in culture collections due to its ability to maintain high microbial survivability (Ge et al., 2024; Morgan et al., 2006), making it unsuitable as a decontamination strategy for food products.

To overcome the limitations of traditional and freeze-drying methods, supercritical carbon dioxide ($scCO_2$) drying has emerged as a disruptive green technology. In this process, water is not removed by vaporization or sublimation but is directly extracted by dissolving it into the supercritical fluid phase of CO_2 (Boumghar, 2014). Operating at mild temperatures (e.g., 35–45 °C), $scCO_2$ protects thermolabile compounds and prevents the structural collapse typical of AD, yielding products with qualities comparable to FD but in significantly shorter times (Zambon et al., 2016). Furthermore, $scCO_2$ acts as a powerful non-thermal microbial inactivation agent. Its unique solvent properties effectively inactivate spoilage microorganisms and pathogens in both liquid and solid foodstuffs (Ferrentino & Spilimbergo, 2011; Garcia-Gonzalez et al., 2007; Santi et al., 2024; Zulli et al., 2025).

While $scCO_2$ drying has been increasingly explored for the dehydration of fruits, vegetables, and herbs, its application to complex animal-derived matrices like seafood remains extremely limited in the current literature (Bernardo et al., 2024, 2025; Zulli et al., 2026). Furthermore, although Tomic et al. (2019) monitored the chemical and quality stability of $scCO_2$-dried apple, to the best of the authors' knowledge, no study has evaluated the long-term microbiological, physicochemical, and sensory shelf-life of $scCO_2$-dried seafood products.

In this context, the present study aims to comprehensively investigate the application of $scCO_2$ drying in seafood over time. Shrimps of the genus *Metapenaeus* were selected as the model matrix due to their commercial availability, high perishability, elevated water activity, and susceptibility to rapid microbial spoilage and structural degradation, which make them particularly suitable for evaluating both dehydration efficiency and microbial inactivation performance. The study therefore evaluates the potential of $scCO_2$ drying as a combined dehydration and decontamination technology, in comparison with conventional air drying (AD) and freeze-drying (FD). We hypothesize that $scCO_2$ drying can simultaneously achieve effective dehydration and microbial inactivation of shrimp, while better preserving physicochemical quality compared to AD and approaching the quality of FD.

The specific objectives were to: (i) compare the physicochemical quality of the dried products, in terms of texture, color, rehydration capacity, and volatile organic compound profile during refrigerated storage at 4 ± 1 °C; (ii) assess process lethality through a microbial challenge test using *Listeria innocua* and *Enterococcus faecium* as surrogate pathogens; and (iii) monitor the microbiological stability of the dried products during ambient storage at 20 ± 1 °C.

2. Material and methods

2.1. Sample preparation

Frozen shrimps (*Metapenaeus* spp.) were provided by Mancin Nadia Srl (Rivà, Italy). According to the product label, the shrimps were wild caught in the Western and Eastern Indian Ocean (FAO fishing areas 51 and 57). Prior to freezing, the shrimps underwent a mild pre-cooking treatment consisting of blanching at 90 °C for 30 s, followed by individual quick freezing (IQF) to preserve product quality. According to the supplier's specifications, the mean nutritional composition per 100 g of product was: protein 12.0 g, fat 0.9 g (of which saturated fatty acids 0.3 g), carbohydrates 0 g, and salt 2.1 g.

A single batch of frozen shrimp was purchased and subdivided into three sub-batches according to the drying technology applied. This approach was deliberately chosen to minimize raw material variability and ensure that observed differences among treatments were attributable to the drying method rather than to inter-batch differences in the raw material. All sub-batches were transported in an isothermal container and stored at −18 °C in sterile bags until further processing. The same batch was used for all shelf-life time points. Control samples (frozen shrimps) were used to compare the quality of the processed products after rehydration. One sub-batch was transported to the Netherlands for supercritical carbon dioxide drying at the industrial facilities of CO2Dry B.V. (Weesp, The Netherlands). A second sub-batch was sent to the University of Trento (Italy) for freeze-drying. The remaining sub-batch was processed by conventional hot-air drying at the University of Padova (Italy). Detailed operating conditions for each drying treatment are reported in the corresponding sections (Sections 2.2–2.4). Before supercritical and hot-air drying, samples were thawed at 4 ± 1 °C for 16 h.

2.2. Supercritical carbon dioxide drying

Drying with supercritical carbon dioxide ($scCO_2$) was conducted using a pilot scale plant with CO_2 recirculation of the company CO2Dry B.V. (Weesp, The Netherlands). The $scCO_2$ plant is mainly composed of two cylindrical vessels, one for the drying (13 cm internal diameter, 10 L of volume) and one for the CO_2 regeneration containing zeolites for water adsorption. For each test, 1.0 ± 0.1 kg of thawed samples were placed inside cylindrical metallic baskets, previously cleaned with ethanol and dried to remove any residual.

The pressurization of the vessel was carried out at a rapid rate to around 60 bar, which is the CO_2 tank pressure and then to 14 MPa in around 12 min with a constant pressurization rate. The CO_2 flow rate during the process was fixed at 700 L/h. Pressure and flow rate were constantly monitored by a back pressure regulator and a flow controller, respectively. During the drying step, which lasted around 15 h, wet CO_2 exiting from the bottom of the drying vessel passes through the regeneration vessel, where a zeolite bed (MS-562, D&F Techniek B.V., Netherlands) physically adsorbs water. Dry CO_2 is then recycled back to the main vessel. After each treatment, wet zeolites were replaced with dried ones. After the desired treatment time, the vessel was depressurized in 10 min. The internal temperature of the drying vessel, controlled by the heat exchanger and drying reactor jacket, was kept at 35 °C.

Challenge tests were carried out in an analogous small scale pilot plant (volume 150 mL, 2.5-cm diameter), at the same conditions of temperature, pressure, time and CO_2 flux, as previously described in Zulli et al. (2026) (Section 2.2).

2.3. Hot-air drying

Hot air drying was employed as a conventional method for comparison with the innovative drying techniques. A commercial convective dryer (Biosec Domus B5, Tauroessicatori, Vicenza, Italy) was used at 60 °C for approximately 6.5 h, until reaching a water activity of the samples similar to the one obtained by $scCO_2$ drying.

2.4. Freeze drying

The lyophilization of shrimps was done using a laboratory-scale freeze dryer (Lio 5P, Cinquepascal srl, Trezzano sul Naviglio, MI, Italy). Frozen shrimps were placed in a freeze-dryer chamber, operating at a temperature of −50 °C. Both the condenser and the drying chamber were connected to a vacuum pump, achieving a vacuum level of 5×10^{-1} mbar during the drying process. These conditions were maintained through the entire drying operation (104 h) until reaching a water activity of the samples similar to the one obtained by $scCO_2$ drying.

2.5. Shelf life studies

For the shelf-life study, dried shrimp samples were vacuum-packaged in multilayer high-barrier films (PE/EVOH/PE; Niederwieser GmbH, Sulzberg, Germany) and stored in the absence of light for up to 4 months. The experimental design involved two distinct storage conditions to assess different stability parameters. Samples intended for microbiological analyses were stored at 20 ± 1 °C to determine safety and microbial behavior under ambient storage conditions. Meanwhile, samples for physicochemical and oxidative analyses were kept at 4 ± 1 °C to evaluate the shelf-life based on chemical quality retention. These two storage conditions were not intended to describe a single integrated shelf-life trajectory. Rather, they were designed to assess two distinct aspects of product stability: microbiological behavior under ambient storage and physicochemical quality retention under refrigerated storage. Additionally, the reference sample consisted of frozen shrimps thawed at 4 ± 1 °C for 16 h prior to analysis. Evaluations were performed every 30 days as described in the following sections.

2.6. Rehydration

Shrimps dried with the three different processes were rehydrated based on the method stated by Akonor et al. (2016). From each drying method, the initial weight of eight dried shrimp replicates was recorded. The samples were then soaked in distilled water, maintaining a sample-to-water ratio of 1:100, and rehydrated for 14 h at a controlled temperature of 4 °C. At the end of this period, excess surface water was gently blotted off with paper towels, after which the final weight of the rehydrated shrimps was measured. Rehydration ratio was calculated according to the following equation:

$$Rehydration\ ratio\ (g/g) = (Rehydrated\ sample\ weight)/(Dried\ sample\ weight) \quad (1)$$

2.7. Chemico-physical analysis

A set of physicochemical analyses was conducted on dried shrimp samples produced by three different drying methods at 0, 1, 2, 3, and 4 months of storage to monitor quality changes over shelf life. The results were compared with reference samples that were frozen and subsequently thawed at 4 ± 1 °C for 16 h prior to analysis. The analyses involved rehydration of the dried samples, followed by the determination of pH, water activity, color, texture, and volatile organic compounds using gas chromatography–mass spectrometry (GC–MS).

2.7.1. pH measurement

For pH measurement, eight rehydrated shrimp samples were chopped and divided into three equal portions. Each portion was weighted and transferred into falcon tubes to obtain three replicates. Deionized water was then added to each tube at a 1:1 (*w/v*) ratio. The mixture was homogenized manually, and pH of the resulting suspension was measured by immersing the electrode of a pH meter (InoLab, WTW GmbH & Co. KG, Weilheim, Germany) (Carneiro et al., 2013).

2.7.2. Water activity

Water activity (a_w) of both reference and dried samples was measured using the instrument Hygropalm HP-23-A (Rotronic). For each measurement, 8 shrimp samples were placed into the machine cup. The measurements were carried out at a controlled temperature of 25 °C. The jar was sealed and the measurement was allowed to equilibrate for 20 min, after which the water activity was recorded. The measurement was then allowed to continue for an additional 5 min. If no change in the displayed value was observed, the analysis was considered complete. If the value varied by more than ±0.005, the measurement was extended in 5 min intervals until stabilization was achieved.

2.7.3. Color

The color of the rehydrated shrimps was measured using a colorimeter (CM 5, Konica Minolta, Singapore) to obtain L* (Lightness/darkness), a* (redness/greenness), and b* (yellowness/blueness) values. The illuminant C was used, and a 2° observer angle was set. The device's probe was positioned on the middle abdominal segment of each shrimp, and measurements were taken from both the front and back sides of eight shrimp replicates. Total color difference (ΔE) was measured using Eq. (2) (Pathare et al., 2013), where values for a_0, b_0, and L_0 were recorded by the color parameters in the reference samples. For each treatment, eight shrimp replicates were tested.

$$\Delta E = \sqrt{\left(L_1^* - L_0^*\right)^2 + \left(a_1^* - a_0^*\right)^2 + \left(b_1^* - b_0^*\right)^2} \quad (2)$$

2.7.4. Texture analysis

Texture analysis was performed using a texture analyzer (TA.XTplus, Stable Micro Systems, Surrey, UK). A single compression test was done with 8 shrimp replicates compressed to 60% strain using a 75 mm plate probe (P/75) at a test speed of 2 mm/s and the trigger force set to 5 g. The sample was positioned at the center of the base plate, with the shrimp oriented so that the head and tail faced the observation window, and the dorsal (back) side was facing the operator (Wang et al., 2018). Hardness (N) was defined as the maximum or peak force reached in the single compression cycle, representing the stiffness of the sample. Resilience was calculated as the ratio of the unloading area to the loading area during the single compression cycle (Dunne et al., 2025).

2.7.5. Volatile organic compounds characterization

Headspace solid-phase microextraction (HS-SPME) combined with gas chromatography–mass spectrometry (GC–MS) was used to extract, concentrate, separate, and identify volatile organic compounds in rehydrated shrimp samples. GC–MS analysis was performed using a Clarus 500 GC system (PerkinElmer AutoSystem XL, Waltham, MA, USA) coupled with a TurboMass Gold mass spectrometer (PerkinElmer) and equipped with a PAL triaxis autosampler (CTC Analytics, Zwingen, Switzerland). The method was adapted from a previously optimized protocol developed for mussels (Bordignon et al., 2024).

Initially, eight shrimps were minced using a knife to obtain a homogeneous sample, from which 1 g subsamples were transferred into three separate 20 mL amber glass vials containing 1 mL of distilled water, 0.5 g of NaCl, and a magnetic stir bar. The mixture was spiked with 50 µL of an internal standard solution (2-octanol 96% GC grade, 0.9875 µg/L in water; Aldrich, Milan, Italy). A 2-cm DVB/Car/PDMS fiber (50/30 µm; Supelco, Sigma-Aldrich, St. Louis, MO, USA) was used for VOCs sampling. Vials were equilibrated for 20 min at 40 °C with continuous stirring before exposing the fiber to the headspace for 60 min

at the same temperature. Volatile compounds were desorbed into the GC injector at 250 °C in splitless mode. Separation was achieved on an HP-Innowax fused-silica capillary column (30 m × 0.32 mm ID, 0.5 μm film thickness; J&W Scientific, Agilent Technologies). Helium served as carrier gas at a flow rate of 1.5 mL/min. The oven temperature program was: 40 °C (3 min), ramped at 4 °C/min to 220 °C (1 min hold), then at 10 °C/min to 250 °C (1 min hold). The transfer line was set at 220 °C. Mass spectra were acquired in electron ionization mode (70 eV) over an *m/z* range of 33–300. Identification was based on spectral matches with NIST 14 and Wiley 7th libraries, and linear retention indices (LRI) were calculated using a C7–C30 n-alkane series under the same chromatographic conditions. GC analyses were performed in duplicate, and volatile compound relative concentrations were expressed as 2-octanol equivalents (ng/mL).

2.8. Microbiological analysis

Thawed and dried samples were placed in 50 mL Falcon tubes and mixed with Ringer solution (Merck KGaA, Darmstadt, Germany) at a weight ratio of 1:9 and vortexed for 90 s at 2400 rpm. Subsequently, serial dilutions (1:10) were performed for the evaluation of total mesophilic count, yeasts and molds, *Pseudomonas* spp., *Enterobacteriaceae* and lactic bacteria, through the standard plate count method. For the enumeration of total mesophilic count, 1 mL of serial dilution was poured into Total Plate count agar (PCA), with a limit of detection of 1 Log CFU/g. Then, the plates were incubated at 30 °C for 72 h. Rose Bengal Agar (RBA) with Chloramphenicol Antibiotic Supplement, Pseudomonas Agar Base (PAB) with Pseudomonas CFC Selective supplement, Violet Red Bile Lactose Agar (VRBLA), and Man, Rogosa, Sharpe agar (MRS) were employed for yeasts and molds, *Pseudomonas* spp., *Enterobacteriaceae* and lactic bacteria, respectively. In these cases, 100 μL of the serial dilutions were spread onto the selective agar, with a limit of detection of 2 Log CFU/g. Then, RBA and PAB plates were incubated at room temperature for 120 h, VRBLA at 37 °C for 24 h and MRS at 37 °C for 48 h under anaerobic conditions. All microbial analyses were conducted under sterile conditions within a laminar flow cabinet (Safemate ez 1.2, Bioair, Siziano, Italy). All media were purchased from Microbial Diagnostici s.r.l. (Cagliari, Italy), while supplements were purchased from Liofilchem s.r.l. (Teramo, Italy).

2.9. Challenge test

A microbial challenge test was performed in accordance with ISO 20976-2:2022 (ISO 20976-2:2022, 2022) to evaluate the behavior of selected surrogate microorganisms during processing and storage. Two reference strains were used: *Listeria innocua* ATCC 3309 and *Enterococcus faecium* ATCC 6057, selected as non-pathogenic surrogates for food safety assessment.

Each strain was individually reactivated in Brain Heart Infusion (BHI) broth (Microbial Diagnostici s.r.l., Cagliari, Italy) and incubated overnight at 37 °C to reach the stationary growth phase. Cell suspensions were then centrifuged and resuspended in ringer solution to obtain a final concentration of approximately 10^8–10^9 CFU/mL, as confirmed by plate counting.

Samples were inoculated by adding the bacterial suspension at 1% (*w*/w) of the sample mass, ensuring homogeneous distribution over the sample surface. A total of 13 test units, each prepared in triplicate, were analyzed, including three inoculated controls and ten inoculated and treated samples.

Microbial enumeration was performed immediately after inoculation and at the end of the experimental treatments. *E. faecium* was enumerated on KF Streptococcus agar (Microbiol Diagnostici, Cagliari, Italy) after incubation at 37 °C for 48 h, while *L. innocua* was enumerated on ALOA agar (Agar Listeria according to Ottaviani and Agosti) (Microbiol Diagnostici, Cagliari, Italy) following incubation at 37 °C for 24 h. Results were expressed as log CFU/g. All microbiological analyses were carried out under aseptic conditions in a laminar flow cabinet.

2.10. Sensory analysis

To analyze the consumer perception of rehydrated shrimp samples produced using the three drying methods, after 1 month of shelf life we compared them with the reference shrimps. A sensory test was conducted in the sensory laboratory of Fondazione Edmund Mach with a consumer panel composed of 80 participants (52.5% female and 47.5% male in the 25–73 age range).

The study was evaluated by the Ethics Committee of the Università degli Studi di Trento (Protocol No. 2024-081ESA), which determined that the research did not require formal ethical approval. All participants provided written informed consent prior to participation, and data were collected and processed anonymously in compliance with the General Data Protection Regulation (GDPR).

The participants, based on visual, olfactory and tactile evaluation, first rated their overall liking of the four samples and then, after a brief training, described each sample according to a Check-All-That-Apply (CATA) questionnaire. The panel evaluated the four anonymously coded samples (for each sample 4 shrimps in a plastic cup covered with lid) presented in a balanced order to each panelist. Overall liking was assessed using a 9-point hedonic scale (1 = dislike extremely, 5 = neither like nor dislike, 9 = like extremely). For the CATA analysis, participants were provided with three predefined lists of visual, olfactory, and tactile attributes shown in Table 1 and were asked to select all descriptors they considered appropriate for the appearance, smell and texture of each sample. At the end, questionnaires were administered to collect socio-demographic data and the purchase intentions.

2.11. Statistical analysis

Data are presented as mean and corresponding standard deviations. A two-way analysis of variance (ANOVA) was conducted to assess the effect of treatment and time. When significant interactions or main effects were found, Šídák's multiple comparison post-hoc test was applied to determine significant differences between specific groups. Statistical significance was set at p-value <0.05. All analyses were performed using GraphPad Prism version 10.3.1 (GraphPad Software, San Diego, CA, USA) and R Studio version 2025.05.1 + 513 (Posit Software, PBC, Boston, MA, USA). It should be noted that each drying treatment was performed as a single run using a single raw material batch. The reported replicates therefore represent analytical subsamples rather than independent process replicates. Consequently, the between-treatment statistical comparisons should be interpreted as indicative of analytical variability within each treatment and future work should include multiple independent drying runs per treatment to assess process reproducibility and validate these findings.

Table 1
Visual, olfactory, and tactile descriptors for Check-all-that-apply sensory test.

Visual	Smell	Texture
Pink	Fresh fish	Soft
White	Ammonia	Firm
Orange	Marine	Flaky
Black	Swampy	Gummy
Gray	Vinegar	Juicy
Brown	Putrid	Fibrous
Pressed	Smoky	Gelatinous
Shrinked	Ferrous	Flaky
Fleshy	Rancid	
Shiny	Odorless	
Smooth	Intense	
Rough	Wet rag	
Dry		

3. Results and discussion

As defined in the experimental design, the three drying processes were carried out to reach a comparable target water activity (a_w) of approximately 0.55 to ensure a fair evaluation of the treatments. However, the time required to achieve this target varied significantly among the techniques. Air drying (AD) required the shortest processing time (approximately 6.5 h), whereas freeze-drying (FD) required the longest, taking approximately 104 h compared to the $scCO_2$ process, which took 15 h. The results are presented according to the two storage conditions used in the experimental design. Physicochemical, structural, and volatile quality changes were evaluated during refrigerated storage at 4 ± 1 °C, whereas microbiological stability was assessed during ambient storage at 20 ± 1 °C. Therefore, trends observed in the physicochemical dataset are not used to infer microbial behavior, and microbiological results are not used to explain chemical changes.

During the 4-month shelf life at 20 °C, a_w remained stable for all dried samples.

Based on these premises, the following sections focus on the comparison of the dried shrimps obtained by three different methods, AD, FD, and $scCO_2$, in terms of microbial safety during storage time of 4 months and quality-related properties, including structural and physicochemical characteristics.

3.1. Rehydration ratio

Rehydration is the process of moistening a dry product and it is considered an important quality parameter as it reflects the extent of structural disintegration that occurs during drying (Akonor et al., 2016). The trends of rehydration ratios of shrimps under different drying methods over 4 months of shelf life are presented in Fig. 1A.

As shown, during the first month of storage, the lowest rehydration ratio was observed in hot-air-dried shrimp, while $scCO_2$-dried samples exhibited values numerically slightly lower than freeze-dried samples but not statistically different ($p > 0.05$). In the second month, the rehydration ratio of $scCO_2$ (2.99 ± 0.52) decreased and became similar to that of AD samples (2.53 ± 0.16). From the third to the fourth month, a consistent trend was observed, with the highest rehydration ratio in freeze-dried samples, followed by $scCO_2$-dried and then hot-air-dried shrimps.

The low value observed in AD can be attributed to protein shrinkage and structural tightening caused by the relatively low drying temperature combined with a slow drying rate. The prolonged drying time reduces porosity and provides more opportunity for protein denaturation and contraction. Consequently, the water channels responsible for transporting water into the muscle fibers become obstructed, resulting in a lower rehydration rate (Akonor et al., 2016; Ersan & Tugrul, 2021). Similar findings were reported by Luo et al., who found that hot air-dried shrimps showed lower rehydration rates in comparison with roasted-dried and microwave-dried shrimps (Luo et al., 2025). For FD samples, as freeze drying causes rapid evaporation during sublimation, the removal of ice crystals creates a highly porous network within the matrix, preventing shrinkage and structural collapse. This porous structure facilitated water penetration, resulting in enhanced water uptake and the highest rehydration ratio throughout the four months of storage. These results are consistent with the findings of Lin et al. (1999), that freeze-dried shrimps showed more rehydration potential than air-dried shrimps. However, $scCO_2$ with intermediate rehydration capacities, showed the values mainly much closer to those of freeze drying. This suggests that $scCO_2$ drying is able to better preserve the shrimp microstructure compared to hot air drying. Overall, these results demonstrate that the drying method strongly influences shrimp microstructure, which determines the extent of water uptake during rehydration.

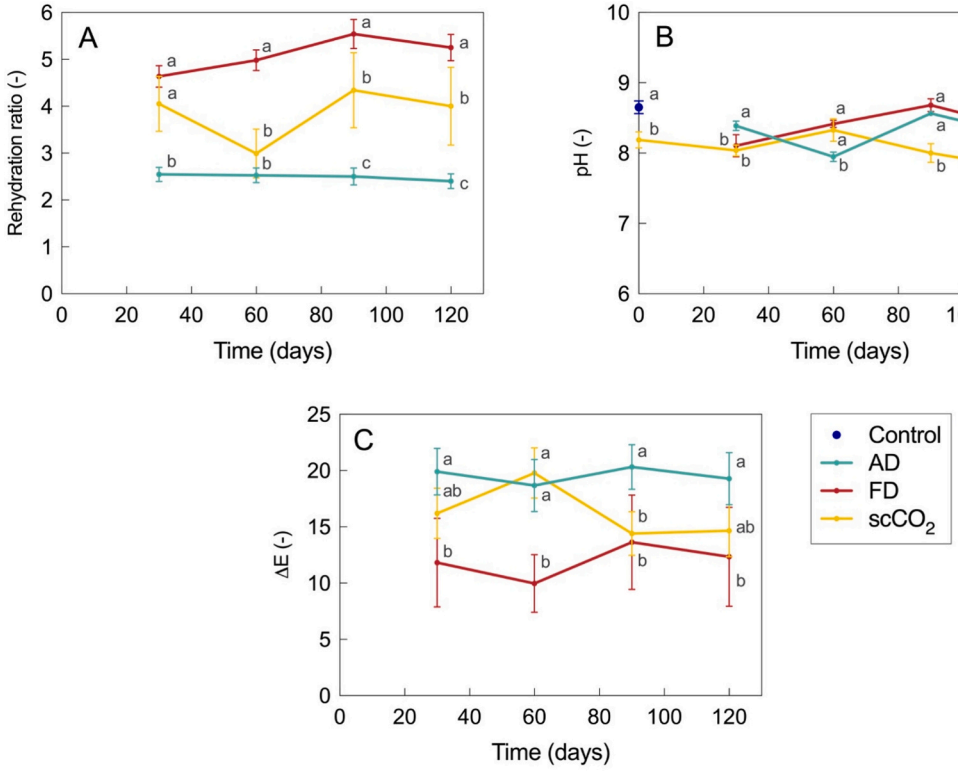

Fig. 1. (A) Rehydration ratio, (B) pH, and (C) total color difference (ΔE) in reference shrimps (CTRL) and in samples dried by hot air (AD), freeze-drying (FD), and supercritical CO_2 ($scCO_2$) and rehydrated during a 4-month shelf life at 4 ± 1 °C. Different lowercase letters (a, b, c) indicate statistically significant differences among treatments at the same time point (p-value <0.05).

3.2. Chemicophysical properties

3.2.1. Color measurement

Since color is the first sensory attribute consumers encounter, it is a critical quality attribute in food products, significantly influencing consumer perception and purchasing decisions. Yanar et al. (2004) reported that the commercial value of shrimp largely depends on its body color, which is primarily determined by astaxanthin, the carotenoid pigment responsible for the red–orange pigmentation of the muscle tissue. Normally, the color of the food products changes during drying due to the non-enzymatic and enzymatic browning that happens in the drying process. L*a*b* color parameters measured in dried samples are represented in Table S1, Supplementary data, ΔE was measured for the samples, measuring their color difference with the thawed shrimps shown in Fig. 1C.

The L* value (lightness) ranges from 0 (black) to 100 (white) and reflects the degree of sample whiteness. Throughout storage, all dried samples exhibited higher lightness than the control (CTRL). Contrary to the findings of Ren et al. (2021), who reported darker color formation in hot-air-dried products due to enhanced Maillard browning, both CO_2-dried and hot-air-dried shrimp in the present study showed comparable L* values and were lighter than the freeze-dried samples during the first two months of storage. Furthermore, in disagreement with (Li et al., 2020), who observed higher L* values in freeze-dried shrimp than in hot-air-dried samples after identical boiling times, our results showed lower lightness for the FD treatment. This behavior may be associated with the highly porous structure developed during freeze drying, which can reduce light reflection and produce a less bright surface, resulting in lightness values similar to those of the $scCO_2$-dried samples.

The a* value, which represents the red–green color axis, increased upon heat exposure as a result of the denaturation of carotenoproteins and astaxanthin release, leading to a more reddish appearance of the shrimp (Akonor et al., 2016). Accordingly, freeze-dried samples generally exhibited lower redness than other samples. During the first two months of storage, the a* values of the CO_2-dried samples were comparable to those of the air-dried samples; however, toward the end of shelf life they decreased and approached the values of the freeze-dried treatment, indicating reduced redness.

The b* value, corresponding to the yellow to blue axis, was higher in air-dried samples, most likely due to non-enzymatic browning and Maillard reactions occurring during hot-air drying. In contrast, CO_2-dried samples showed lower b* values, which were statistically similar to those of the freeze-dried samples.

As shown in Fig. 1C, all samples exhibited ΔE values greater than 3.5 relative to thawed shrimps, indicating a clearly perceptible color difference (Zambon et al., 2020). Freeze drying resulted in the lowest color difference from control shrimps due to the minimal color changes at low temperature. The ΔE values decreased until the second month and then increased toward the end of storage, a trend also observed for AD samples. However, air drying showed the highest ΔE, confirming the occurrence of more intense Maillard and browning reactions, as well as protein denaturation which releases astaxanthin at higher temperatures combined with more drying time. The $scCO_2$ samples exhibited ΔE values similar to those of freeze-dried shrimp, except in the second month, and overall demonstrated a better capacity to preserve shrimps' quality than air drying.

3.2.2. pH measurement

As shown in Fig. 1B, the blue dot represents the pH of thawed shrimp (8.65 ± 0.09). During storage, hot-air drying did not lead to a substantial reduction in pH. Although a temporary decrease was observed in AD in the second month, the pH increased again during the remaining storage period and approached the value of the thawed samples. In contrast, $scCO_2$ drying resulted in a significant reduction in pH from the initial time point and showed an overall decreasing trend throughout storage. The pH values of $scCO_2$-dried samples were comparable to those of freeze-dried samples during the first two months and became even lower in the later stages, reaching a final value of 7.77 ± 0.13 at the end of month four of storage. According to Bai et al. (2024), where they measured pH values of dried shrimps, high-quality dried shrimp should have pH values below 8.10, whereas pH values above 8.35 indicate low quality.

Under refrigerated storage at 4 ± 1 °C, the lower pH observed in the $scCO_2$-dried samples at the end of storage compared with AD (8.23 ± 0.06) and FD (8.33 ± 0.06) suggests a lower accumulation of alkaline compounds associated with quality deterioration. However, the acidic effect associated with residual CO_2 after treatment may also have contributed to the observed pH values. Therefore, while the results suggest that $scCO_2$ treatment may help maintain product quality during storage, pH values should be interpreted in combination with microbial and other spoilage-related analyses when evaluating spoilage development which is further discussed in Section 3.3.

3.2.3. Volatile organic compounds characterization

Volatile organic compounds responsible for odors and flavors are important quality features of dried shrimp which can directly influence consumer acceptance. HS-SPME GC–MS was used to determine the types of VOCs and their relative content in rehydrated shrimps, comparing them with thawed shrimps (CTRL). The VOCs change the most and their intensity is shown in the heatmap in Fig. 2A. A total of 30 volatile compounds with the most variance among methods of drying were identified across the samples, classified into 8 groups including aldehyde (4), ketone (2), alcohol (11), hydrocarbon (7), heterocycle (2), amine (2), acid (1), sulfur compound (1). The graph shows that the drying method significantly affects the volatile compound profile, as indicated by different letters. At the beginning of shelf life, VOC profiles were similar among drying methods, but by the end (M4) significant differences appeared. $scCO_2$ and FD samples showed similar profiles and contained fewer volatile compounds than hot air–dried samples.

Aldehydes, the dark green segment, are important aroma molecules as they have a low odor threshold and they can strongly affect the odor and flavor of dried marine products (Hu et al., 2021). Aldehydes may originate from different chemical pathways. In particular, they are commonly associated with Strecker degradation of amino acids, whereas linear aldehydes, such as pentanal, hexanal, and nonanal, are frequently reported as volatile compounds consistent with the degradation of unsaturated fatty acids. In the present study we observed a small amount of them present in CTRL, as shown in Fig. 2C. 3-Methyl-butanal, having a burnt sweet and roasted aroma, is produced as a Maillard intermediate in Strecker degradation of leucine (Zheng et al., 2025). This compound is higher in AD dried shrimps, confirming the Maillard reaction and higher time of drying in this procedure (Luo et al., 2025). In contrast, in the freeze-dried samples in the first two months the concentration of 3-methyl-butanal was the lowest as there is no high temperature in this drying procedure. However, in months 3 and 4, this compound was equally present in $scCO_2$ and FD showing the better preservation of shrimp quality in CO_2 drying. Our observations are in agreement with the conclusions drawn by Sun et al. (2022), who found that 3-Methyl-butanal contributed more to the aroma of hot-air dried shrimps. Pentanal (fruity and bakery like aroma) and hexanal (raw grassy and greasy aroma), consistent with thermal decomposition and oxidation of linoleic acid (Hu et al., 2021; Zheng et al., 2025) which were mostly greater in $scCO_2$ dried shrimps in the first three months in comparison with FD, however it became the same as FD at the end of shelf life. Moreover, nonanal (fatty aroma) was different between samples only at the end of shelf life.

Ketones, which are mainly lipid oxidation products, have a high odor threshold, thus they may contribute less to odor and flavor of the shrimps compared to aldehydes and alcohols (Luo et al., 2025). The only significant difference observed was in 2-nonanone, that could cause creamy and fruity flavor (Sun et al., 2022), and 2-propanone (sweet aroma), which present generally in very little amounts in FD and $scCO_2$,

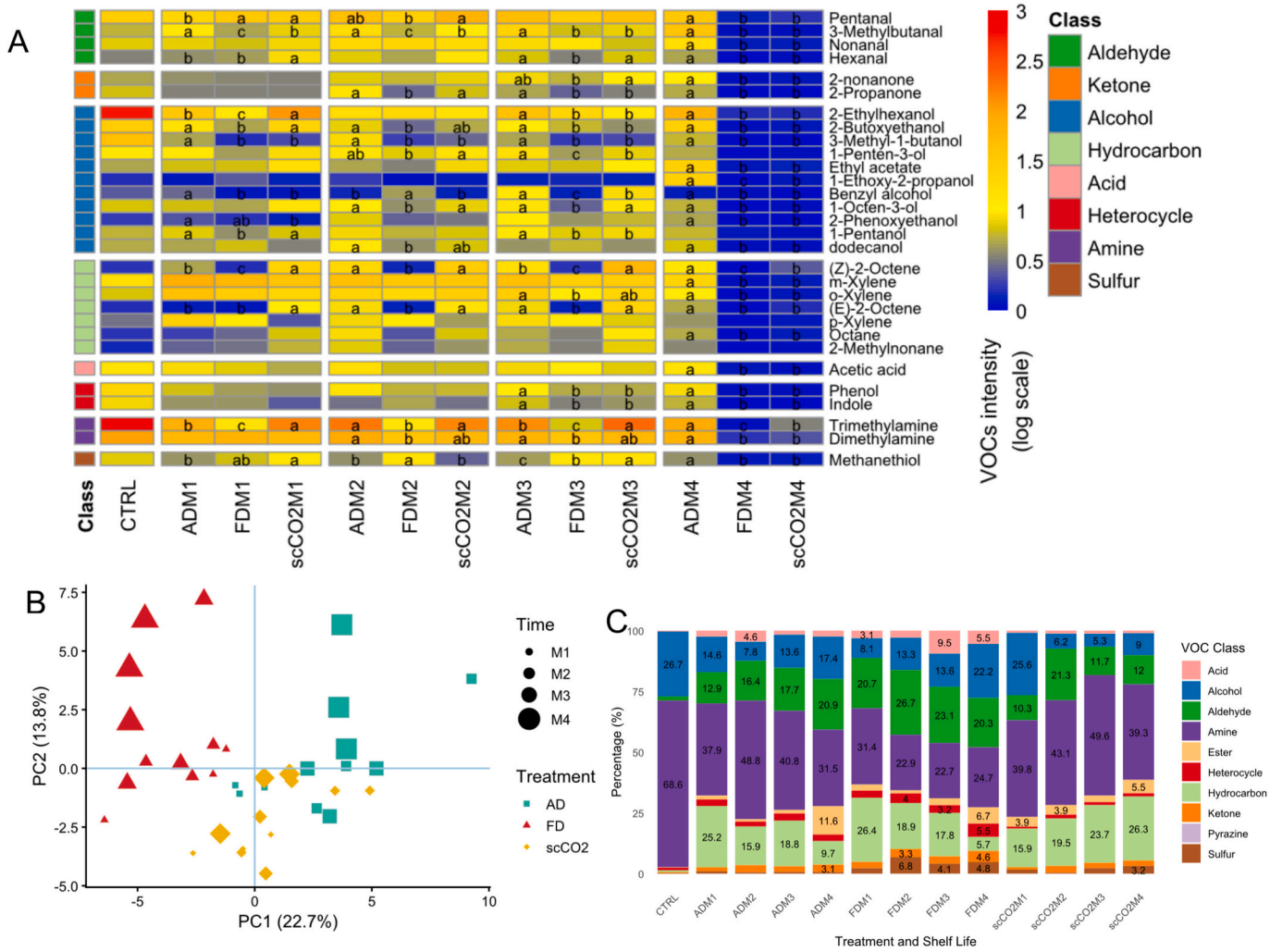

Fig. 2. (A): Heatmap of VOCs in the control sample (CTRL) and dried treatments during four months of shelf life at 4 ± 1 °C (M1–M4). Each row represents one of the 30 VOCs showing the greatest variation among samples, while each column corresponds to the drying method and storage time (M1, M2, M3, and M4). The color scale represents the relative intensity of each compound (log scale), with deeper red indicating higher intensity and blue indicating lower intensity. Differences among drying methods are indicated by different letters, whereas the absence of letters indicates no significant differences between treatments. (B): Principal component analysis score plot of VOCs across different drying methods and storage times. Treatment shape size increases from month 1 to month 4. (C): Stacked Bar chart showing the distribution of VOCs classes in shrimp samples dried by different methods over four months of storage.

and in higher amounts in AD in month four, indicating oxidation occurred in the convective oven drying.

Alcohols with higher odor threshold, have a mild effect on the final odor and flavor and are mainly generated by the reaction of lipoxygenase on unsaturated fatty acids or the reduction of carbonyl compounds (Aprea et al., 2018; Mall & Schieberle, 2017). There is a fluctuation between the contents of alcoholic compounds in different dried shrimps including 1-octen-3-ol with mushroom and fishy aroma (Sun et al., 2022). The most abundant alcohol in thawed shrimps was 2-ethylhexanol which was also the highest in AD during shelf life and decreased significantly until month 4 in FD and $scCO_2$.

Regarding amines, compounds such as trimethylamine are produced from the thermal decomposition of trimethylamine oxide, a small organic compound found in marine animals, and together fish and ammonia odors are used for monitoring freshness in aquatic products. Due to this it is the highest in control samples (68.6% of the total VOCs) and present in lower amounts in dried ones as shown in Fig. 2C (Sun et al., 2022). During shelf life, trimethylamine was highest in $scCO_2$, followed by AD and then FD. However at the end of shelf life it became the same in FD and $scCO_2$, and they were significantly lower than in AD, suggesting AD shrimps were expected to exhibit a more intense fishy odor (Zheng et al., 2025). The result agreed with the findings of Sun et al., who stated that amines, most importantly trimethylamine, increased with more heat processing (Sun et al., 2022).

The remaining VOCs consisted of sulfur-containing compounds which have low odor threshold. In shrimps, methanethiol can be generated by the degradation of the sulfur-containing amino acid, methionine, present in muscle proteins. In FD samples, sulfur compounds contributed more to their odor and flavor especially in month 2, 3 and 4 as shown in the brown sections in Fig. 2C.

Fig. 2B shows the PCA score plot of VOCs for both treatment and time of shelf life. The PCA score plot suggests that the different drying methods are associated with distinct changes in volatile composition during shelf life. The first two components explain 36.5% of the total variance and should therefore be interpreted as an exploratory visualization of the data structure. PC1 (22.7%) is mainly distinguishing FD and AD samples, both of which showing greater variability during shelf life compared to $scCO_2$ samples which show a more similar profile during the time. These results are consistent with the VOC classes represented in Fig. 2C, as FD treatments showed the highest variability in VOC classes, followed by AD, while $scCO_2$ treatments had more stable VOC composition.

A minor overlap is seen between the AD and $scCO_2$ treatments, mainly between months 3 and 4, revealing some degree of similarity in the VOCs generated or remaining in these drying methods. This separation supports the conclusion that different drying methods of shrimps influenced the formation or retention of volatile compounds through multiple possible pathways, including Maillard/Strecker reactions, thermal degradation, and lipid-derived volatile formation. However, the specific contribution of lipid oxidation cannot be confirmed from VOC data alone and should be verified in future studies using direct oxidation indices.

The stacked bar chart in Fig. 2C illustrates the relative percentage distribution of VOC classes across different treatments and shelf-life conditions. The volatile constituents before and after drying were mainly dominated by amines, specifically trimethylamine with fishy odor, followed by alcohols and aldehydes. Hydrocarbons also showed relatively high proportions in several treatments. However, due to their high odor thresholds, hydrocarbons are generally not considered major contributors to flavor but mainly serve as precursors for the formation of other important aroma compounds. Pyrazines were detected only in thawed shrimp samples, including 2,5-dimethylpyrazine, which is associated with chocolate-like and creamy aromas, and 2,5-dimethyl-3-ethylpyrazine. Previous studies have reported that pyrazine content increases primarily during roasting rather than drying of shrimp meat, as roasting enhances Maillard reactions promoted by reduction of water activity (Duppeti et al., 2022).

3.2.4. Texture analysis

Shrimp texture is another important quality attribute affecting consumer sensory perception and acceptance. To compare the effect of drying method and shelf-life period on the mechanical firmness of shrimp samples, a single compression test was performed. Two parameters were obtained from the force–time curve: hardness and resilience. Hardness (N) was defined as the maximum force, representing the stiffness of the sample. Resilience reflects the elastic behavior of the material after deformation, showing the ability of a material to recover its original shape once the applied force is removed. Higher resilience values indicate greater elastic recovery, whereas lower values indicate increased plastic deformation of the sample structure (Dunne et al., 2025).

As shown in Fig. 3A, the drying method significantly influenced the texture evolution of shrimp during storage. In the first month, no notable differences in hardness were observed among the treatments, all showing a close hardness to control samples. However, over the storage, $scCO_2$-dried shrimp generally exhibited the highest hardness values, whereas freeze-dried samples showed the lowest hardness, indicating a softer texture. The hardness of FD samples was comparable to that of hot-air-dried shrimps for most of the storage period, except in the second month, when AD samples showed the highest hardness values among all treatments. This can be attributed to the more porous structure formed during sublimation in FD and more densified structure formed after CO_2 drying. The present results are in contrast with the findings reported by (Zambon et al., 2020) who detected no significant difference in hardness between $scCO_2$-dried and freeze-dried red pepper samples. In the current study, however, these drying methods produced significantly different hardness values, which may be attributed to differences in structural characteristics between plant tissues and the fibrous muscle matrix of shrimp. Our results are consistent with the outcomes of texture sensory CATA test, in which AD and $scCO_2$ dried shrimps were defined firmer than freeze-dried and thawed shrimps which were perceived softer and juicier by the panelists.

In terms of elasticity, generally FD samples were less elastic than other dried shrimps. This may be because of the formation of larger pores during the sublimation process in freeze drying. In other words, the high porosity and more swelling after rehydration in freeze-dried samples loosen the bindings in the structure and reduce the elasticity of the tissue. These findings support earlier findings of Phahom et al., who reported that springiness in crab stick by-product dried with the freeze drying method was lower than that dried at 50 °C and 60 °C hot air (Phahom & Soubsub, 2022). Moreover, Guiné and Barroca obtained that freeze drying in mushrooms and onions led to a noticeable decrease in springiness despite the high porosity, suggesting a brittle texture caused by the porous matrix formed during sublimation (Guiné & Barroca, 2011).

$ScCO_2$ and hot-air-dried shrimps exhibited greater elastic behavior during storage, indicating better preservation of textural properties. This finding was consistent with the sensory CATA texture evaluation (§3.4), where $scCO_2$-dried samples, followed by AD samples, were more frequently perceived as chewy compared with the other treatments.

3.3. Microbiological safety

The microbial quality of thawed and dried shrimp samples during storage at 20 °C for 4 months is reported in Fig. 4, which shows the evolution of total mesophilic bacteria, yeasts and molds, and *Pseudomonas* spp.

The thawed shrimp (CTRL) exhibited an initial microbial load of 5.44 ± 0.08 Log CFU/g for mesophilic bacteria and 5.83 ± 0.22 Log CFU/g for *Pseudomonas* spp., while yeasts, molds, *Enterobacteriaceae*, and lactic acid bacteria were under the detection limit at day 0.

Drying treatments resulted in significantly different microbial reductions. $ScCO_2$ induced the strongest microbial inactivation already at day 0, reducing the mesophilic count to 1.57 ± 0.62 Log CFU/g, while

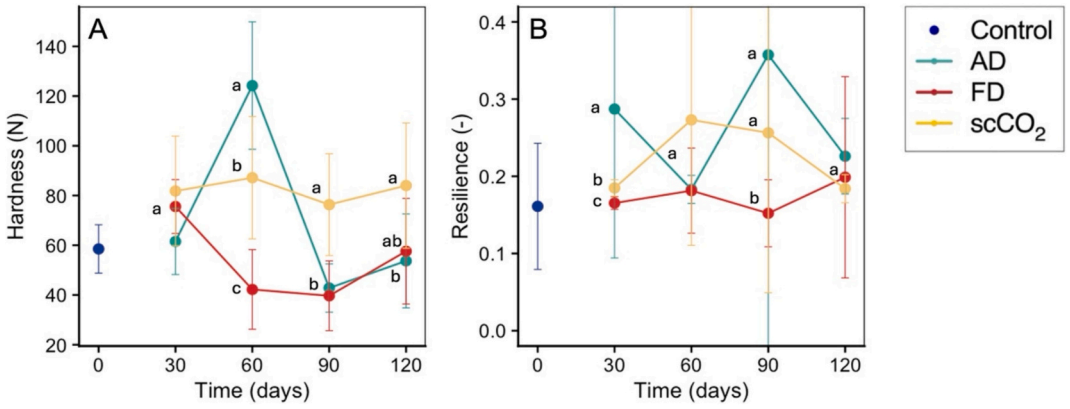

Fig. 3. (A) Hardness (N) of shrimps dried by different drying methods, (B) Resilience, indicating the elastic behavior of shrimp dried using three different methods compared with reference shrimps (CTRL), evaluated during four months of shelf life at 4 ± 1 °C (M1–M4).Different lowercase letters (a, b, c) indicate statistically significant differences among treatments at the same time point (p-value <0.05).

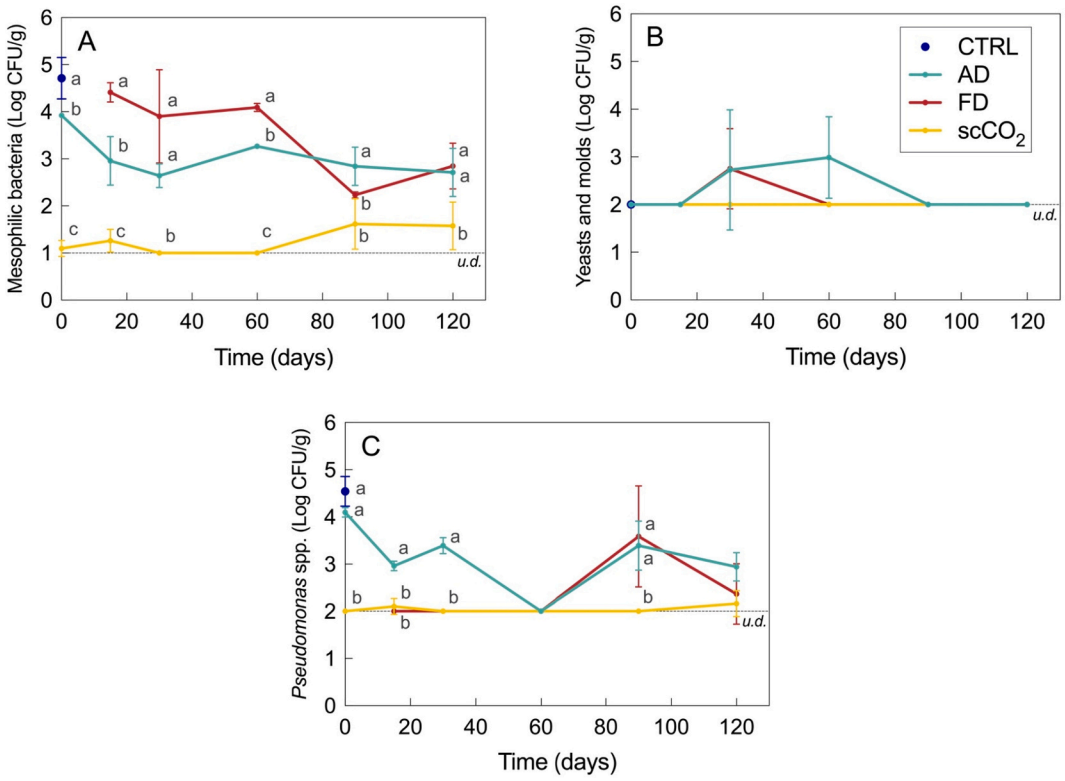

Fig. 4. Microbial load of (A) mesophilic bacteria, (B) yeasts and molds, and (C) *Pseudomonas* spp. in thawed shrimps (CTRL) and in samples dried by air (AD), freeze-drying (FD), and supercritical CO_2 ($scCO_2$) shrimps during a 4-month shelf life at 20 °C. Different lowercase letters (a, b, c) indicate statistically significant differences among treatments at the same time point (p-value <0.05). *u.d.*: under detection limit, 1 Log CFU/g for mesophilic bacteria, 2 Log CFU/g for yeasts and molds and *Pseudomonas* spp.

Pseudomonas spp. and yeasts and molds were below the detection limit (< 2 Log CFU/g). In contrast, hot-air drying (AD) led to a limited reduction, with mesophilic bacteria decreasing by approximately 1 Log unit compared to the control sample. This outcome is consistent with conventional thermal drying at moderate temperatures (e.g., 60 °C), which is generally insufficient to achieve pasteurization. Microbiological data for freeze-dried (FD) samples were not available at day 0 due to practical constraints; the first measurement for FD samples was therefore performed at day 30. As a consequence, no direct quantitative comparison of microbial inactivation efficacy immediately after processing can be made between FD and the other two treatments. However, results from subsequent sampling points indicated limited or negligible inactivation of mesophilic bacteria, while a substantial reduction of *Pseudomonas* spp. was observed. These findings align with the widely accepted principle that freeze-drying does not lead to a significant microbial reduction. The limited reduction in total mesophilic bacteria is expected, as many Gram-positive bacteria are highly resistant to the dual stresses of freezing and desiccation (Morgan et al., 2006). Conversely, the substantial reduction of *Pseudomonas* spp. can be attributed to the higher structural susceptibility of Gram-negative bacteria, more vulnerable to the mechanical stress of ice crystal formation and osmotic shock during sublimation (Palmfeldt et al., 2003). *Enterobacteriaceae* and lactic acid bacteria remained under the detection limit in both thawed and dried samples throughout the entire storage period (data not shown).

Overall, the results at the beginning of storage clearly indicate that $scCO_2$ drying was the most effective process in terms of microbial inactivation, highlighting its potential to improve the microbiological safety of dried shrimp products.

During shelf life, $scCO_2$-dried samples consistently exhibited the lowest microbial loads, confirming their superior microbiological stability. Total mesophilic bacteria remained close to the detection limit throughout storage, whereas AD and FD samples showed higher microbial counts at most time points. Yeasts and molds were sporadically detected in AD and FD samples between 30 and 60 days, while they remained undetected in $scCO_2$-dried samples over the whole storage period. Similarly, *Pseudomonas* spp. were never detected in $scCO_2$-treated samples, whereas they persisted at higher levels in AD samples and were detected after 90 days in FD samples.

To further investigate the microbial inactivation potential of $scCO_2$ drying against pathogenic bacteria, a challenge test was conducted using *Listeria innocua* and *Enterococcus faecium* as surrogate microorganisms, to verify the antimicrobial efficacy of this innovative technology. Prior to inoculation, all samples were microbiologically screened to confirm the absence of *Listeria* spp. and *Enterococcus* spp., ensuring that the observed microbial reductions were solely attributable to the experimental challenge and subsequent $scCO_2$ treatment.

For *L. innocua*, control samples showed average initial concentrations ranging between 5.95 and 6.95 Log CFU/g. After $scCO_2$ treatment, *L. innocua* was below the detection limit in all treated samples, corresponding to a log reduction ≥3.95–4.95 Log CFU/g, depending on the replicate.

Similarly, *E. faecium* exhibited initial loads of 6.44–6.68 Log CFU/g in control samples. After $scCO_2$ drying, *E. faecium was* detected in only 1 out of 10 treated samples for each test, with residual populations ranging between 2.03 and 2.14 Log CFU/g. This corresponded to an overall microbial reduction of ≥4.38–4.54 Log CFU/g.

These results demonstrate a strong inactivation capacity of supercritical CO_2 drying against both Gram-positive surrogate microorganisms, confirming its effectiveness not only in reducing natural spoilage flora but also in significantly lowering populations of bacteria relevant to food safety. Similar pasteurization effects for *L. innocua* and other pathogen-surrogate bacteria using $scCO_2$ were also obtained by $scCO_2$ drying on other matrices, both non-protein based, such as strawberries

(Zambon et al., 2022), and protein matrices, like chicken breast (Morbiato et al., 2019).

It should be noted that the challenge test was conducted at small scale (150 mL vessel) under the same operating conditions as the full-scale process (temperature, pressure, treatment time, CO_2 flux, and matrix dimension). Scale-up validation on the full 10 L industrial vessel remains an important objective for future work, as differences in CO_2 diffusion geometry may affect lethality outcomes.

3.4. Sensory analysis after one month of storage

The appearance, odor and tactile-based liking scores presented in Fig. 5A indicate that the control shrimp samples received similar preference scores to freeze-dried samples and were generally preferred more by consumers. This similarity may be attributed to the freeze-drying process, which removes moisture through sublimation at low temperatures, therefore minimizing thermal degradation and structural damage. Previous studies have shown that vacuum freeze drying preserves the microstructure of food matrices, producing a more porous structure and reduced volume shrinkage because of the absence of significant heat exposure and oxygen (Ratti, 2001).

In contrast, the hot-air-dried and CO_2-dried samples received comparable liking scores in terms of appearance, odor and texture from the panelists, showing that they are similar in sensory acceptability.

CATA analysis was used to assess visual, olfactory, and texture attributes to better understand the differences between dried shrimp samples for each descriptor. In general, the greatest differences among the samples were found in visual attributes, followed by textural properties, whereas olfactory attributes exhibited the least variation.

Fig. S1 (Supplementary data), shows the pictures of dried and rehydrated shrimps given to panelists. It was observed that air drying caused the highest density and shrinkage resulted in more deformed shrimps with more orangish color at the surface, while in FD samples the shape was much more preserved and in CO_2 dried shrimps the extension of shrinkage was more controlled than AD, with more whitish surface. In supercritical CO_2 drying, the absence of a liquid–vapor interface eliminates surface tension effects, therefore capillary stresses that typically cause structural collapse during conventional air drying are prevented (Tomic et al., 2019).

Regarding the visual descriptors (Fig. 5B), thawed and freeze-dried shrimp were predominantly described as pink, fleshy, and shiny, with these attributes being more frequently reported in thawed samples than in freeze-dried ones. In contrast, CO_2-dried shrimp were mainly associated with a whitish appearance, while hot air-dried samples were characterized as significantly more orange compared to all other treatments. Both $scCO_2$ and AD were also perceived as more shrinked in

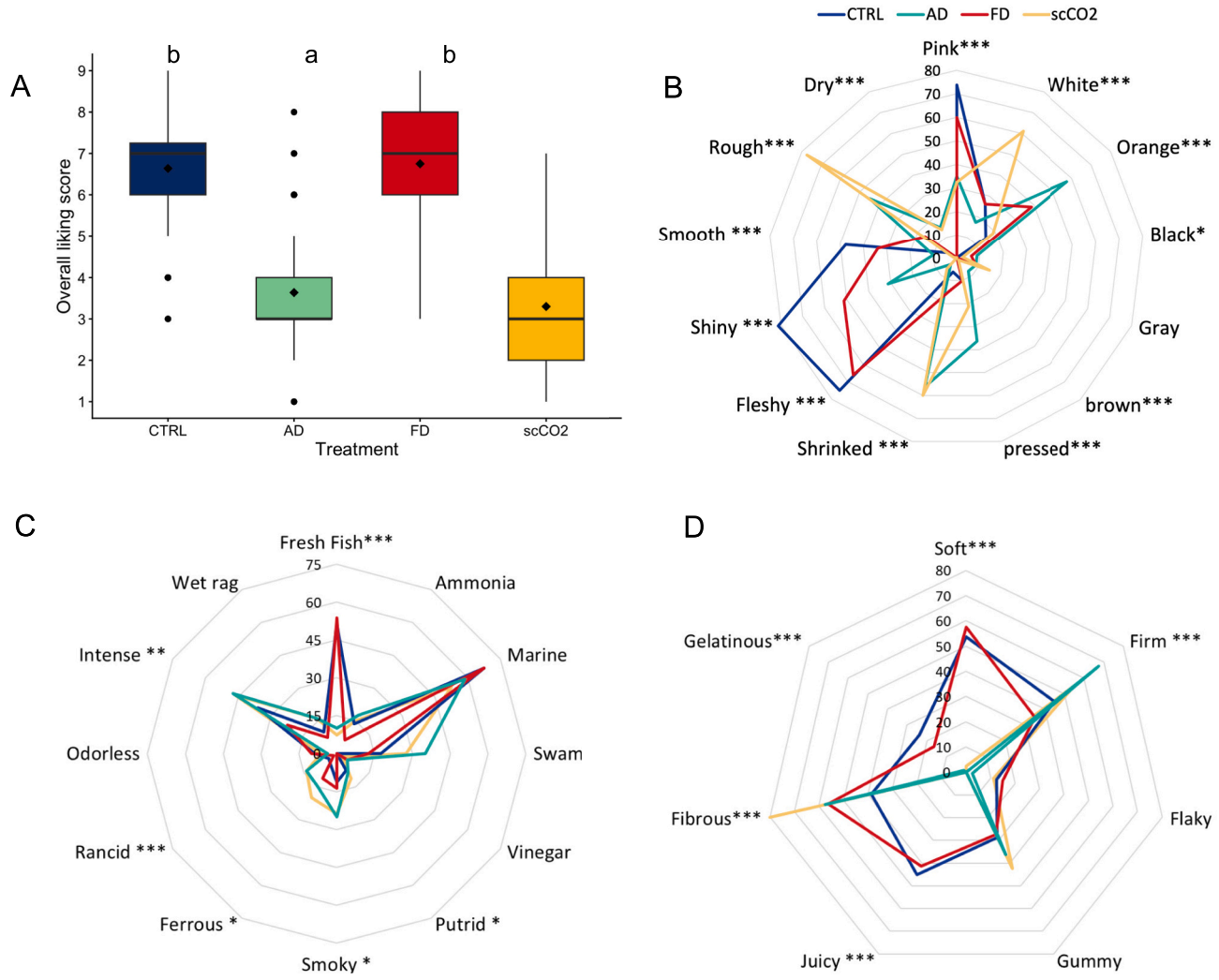

Fig. 5. (A): Appearance, odor and tactile-based liking score box plot of dried shrimps (FD, AD and $scCO_2$, after rehydration) and reference sample (CTRL) at one month of shelf life (M1). (B),(C),(D): Spider charts of the four product profiles based on respectively visual (B), odor (C)and texture (D) CATA descriptors *Stars indicate significant differences among samples based on Cochran's Q test (*$p < 0.05$, **$p < 0.01$, ***$p < 0.001$).

terms of visual texture, with CO_2-dried samples considered rougher than AD dried shrimps. This is consistent with our hardness evaluation in texture results which was higher in $scCO_2$ than AD.

As for olfactory attributes in Fig. 5C, the differences between the odors of dried shrimps were less significantly different among samples than visual descriptors. All samples were smelled marine like, however only thawed and freeze-dried ones were sensed as fresh fish. Swampy off-odor was smelled more in hot-air dried shrimps mainly due to slow drying, followed by $scCO_2$, thawed and FD shrimps. CO_2- and air-dried shrimps were both perceived as rancid, despite exhibiting lower levels of aldehydes at the first month of storage (Fig. 2C), when the sensory evaluation was conducted. This may be due to the higher levels of amines in $scCO_2$, as these compounds are associated with fishy and ammonia-like odors, which can be collectively interpreted as rancid by panelists.

For the CATA texture analysis in Fig. 5D, freeze-dried rehydrated and thawed shrimp were predominantly perceived as soft, juicy, and gelatinous. This is related to the limited protein shrinkage and denaturation during freeze-drying, which preserves the porous structure of the shrimp, allowing for efficient water absorption and resulting in a texture similar to control samples. In contrast, AD and CO_2-dried shrimps were perceived as harder in texture. The gummy or rubbery attributes, however, were similarly perceived across all samples. These findings are consistent with the instrumental texture analysis, which also indicated increased hardness in CO_2-dried samples.

Penalty analysis presented in Fig. S2 was also done to identify the sensory attributes with the greatest impact on consumer liking. The results showed that fleshy, soft, and juicy attributes had the strongest positive effects on liking, indicating that these descriptors are key drivers of product acceptance. Other attributes such as fresh fish aroma, smooth texture, shiny and pink appearance also contributed positively, although to a lesser extent. In contrast, several attributes were associated with significant decreases in liking, with dry, rough, putrid, shrinked, and rancid aroma showing the largest negative impacts. These results emphasize the need to optimize desirable sensory characteristics and reduce negative attributes in CO_2-dried shrimp to enhance consumer acceptance and bring their quality closer to that of freeze-dried products.

Although AD and $scCO_2$-dried samples received similarly lower sensory scores compared with FD samples, the reasons may differ between the drying methods. In AD samples, the lower scores may be associated with muscle shrinkage and possible surface hardening and hard shells caused by the higher drying temperature and longer heat exposure, which negatively affected appearance and texture (Cheng et al., 2021). The lower liking scores observed for $scCO_2$ dried shrimps may be associated with their textural properties, particularly increased hardness, which was shown in texture analysis, as well as the whitish surface color. Although other physicochemical analyses indicated that CO_2-dried samples exhibited intermediate properties and, in some cases, outperformed hot-air-dried samples, lack of juiciness and softness in $scCO_2$-dried shrimps likely reduced consumer acceptance, as also confirmed by the penalty analysis shown in Fig. S2. These results are in contrast to the previous studies as they reported comparable sensory quality between supercritical CO_2-dried and freeze-dried apple slices, although freeze drying generally maintained a slightly superior appearance (Djekic et al., 2018). Overall, although CO_2-based drying technologies are considered promising alternatives due to their potential advantages in microbial reduction, the sensory results indicate that further optimization is required to improve their sensory performance.

In contrast to previous studies on supercritical CO_2 drying of red bell pepper and apples, where texture was well preserved but defects such as loss of color, reduced flavor intensity, hay-like odors for red pepper and surface cracking, and discoloration were reported for apples (Djekic et al., 2018; Tomic et al., 2019; Zambon et al., 2020), the present study for use of CO_2 drying for shrimps showed different outcomes. In our case, color, aroma, and rehydration properties of CO_2-dried shrimp were better preserved compared to air-dried samples. However, texture remained the main limitation shown in the sensory profile, revealing that further optimization is needed to improve the structural properties and achieve a texture more comparable to thawed shrimps.

4. Conclusion

The present study provided the first comprehensive evaluation of the long-term stability of a seafood matrix, namely shrimp, dehydrated via supercritical carbon dioxide ($scCO_2$) and compared with hot-air and freeze-drying technologies. The results demonstrated that $scCO_2$ drying functions as an effective simultaneous dehydration and microbial inactivation method, showing two condition-specific advantages: microbial stability during ambient storage at 20 ± 1 °C, and favorable physicochemical quality retention during refrigerated storage at 4 ± 1 °C. Moreover, a strong antimicrobial efficacy against pathogen-surrogate bacteria was demonstrated in a small-scale challenge test. Because microbiological and physicochemical analyses were conducted under different storage temperatures, these results should be interpreted as complementary but separate assessments rather than as a single integrated shelf-life trajectory. Physicochemically, $scCO_2$ showed color preservation and rehydration capacity intermediate between AD and FD, with values generally closer to those of freeze-dried samples, while maintaining a more stable VOC profile and lower pH than AD. However, $scCO_2$ produced a significantly harder texture and a whitish appearance which led to lower appearance-, odor- and touch-based liking scores compared with FD. Some limitations of the study should be acknowledged such as the multi-site design, which introduced potential confounding factors that were minimized through shared protocols, personnel exchange, and the use of a single raw material batch, as well as the sensory evaluation which was limited to a single time point and to visual, olfactory, and tactile assessment. Future studies should also consider nutritional aspects, particularly fatty acid profiling and astaxanthin quantification, to provide a more comprehensive assessment of the processing potential of $scCO_2$ drying for seafood. In summary, $scCO_2$ drying represents a promising alternative drying technology for seafood, offering superior microbiological safety under ambient storage conditions and favorable physicochemical stability under refrigerated storage conditions; nevertheless, further optimization should focus on sensory attributes, lipid oxidation, and nutritional composition to better meet final market expectations.

CRediT authorship contribution statement

Riccardo Zulli: Writing – review & editing, Writing – original draft, Visualization, Validation, Methodology, Investigation, Formal analysis, Conceptualization. **Faezeh Khoobbakht:** Writing – review & editing, Writing – original draft, Visualization, Validation, Methodology, Investigation, Formal analysis. **Margherita Guidi:** Methodology, Investigation, Formal analysis. **Elena Santoro:** Writing – review & editing, Supervision, Methodology, Funding acquisition, Conceptualization. **Alessandro Zambon:** Writing – review & editing, Supervision, Methodology, Funding acquisition, Conceptualization. **Gerard Hofland:** Writing – review & editing, Supervision, Methodology. **Valerio Giaccone:** Writing – review & editing, Supervision, Methodology, Conceptualization. **Eugenio Aprea:** Writing – review & editing, Visualization, Validation, Supervision, Methodology, Investigation, Funding acquisition, Formal analysis, Conceptualization. **Flavia Gasperi:** Writing – review & editing, Visualization, Validation, Supervision, Methodology, Investigation, Funding acquisition, Conceptualization. **Sara Spilimbergo:** Writing – review & editing, Visualization, Validation, Supervision, Resources, Project administration, Methodology, Investigation, Funding acquisition, Conceptualization.

Ethical statement

The sensory study was evaluated by the Ethics Committee of the Università degli Studi di Trento (Protocol No. 2024-081ESA), which determined that the research did not require formal ethical approval as it did not involve risks to participants. All procedures complied with relevant data protection regulations (GDPR). Participants were informed about the study and provided written informed consent prior to participation. Data were collected and processed anonymously.

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Declaration of competing interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: Spilimbergo Sara reports financial support was provided by Social European Fund Plus, Veneto region and the European Union. Spilimbergo Sara reports financial support was provided by Department of Industrial Engineering (University of Padua). If there are other authors, they declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Co-author Gerard Hofland is affiliated with CO2Dry B.V., which provided the $scCO_2$ drying facility and technical support for this study. All sample analyses were performed independently by academic partners, with no involvement of CO2Dry B.V. in data collection, analysis, or interpretation. The other authors declare that they have no known competing financial or personal interests that could have influenced the work.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.foodchem.2026.150663.

Data availability

Data will be made available on request.

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