

Gaseous Ozone Decontamination of Natural Cocoa Press Cake

Mechanistic constraints, microbiological validation, oxidative quality, and a spatially resolved industrial research framework

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Abstract

Natural cocoa press cake is a compact, heterogeneous intermediate produced by expressing cocoa butter from cocoa liquor. Its position before powder milling makes it an attractive candidate for a targeted antimicrobial intervention. Gaseous ozone (O_3) is a potent oxidant, but antimicrobial activity observed in other low-moisture foods cannot be transferred directly to cocoa cake. The critical barriers are restricted gas access, rapid oxidant consumption by the food matrix, nonuniform microbial localization, and potential oxidative changes in lipids, polyphenols and volatile compounds. This review critically distinguishes established evidence from proposed cocoa-specific hypotheses and develops a spatially resolved validation framework coupling fragment geometry, locally delivered ozone exposure, microbial lethality and product-quality constraints. The proposed sequence is pressing, controlled fragmentation, ozone contact, residual ozone destruction, and hygienic milling. A staged screening and confirmatory validation plan is provided. Currently available evidence supports investigation but does not establish a universally validated ozone lethality process for cocoa press cake. Commercial application requires matrix-specific challenge studies, quality testing, industrial hygienic design and jurisdiction-specific regulatory assessment.

KEYWORDS: *Cocoa press cake; ozone; gas–solid reaction; low-moisture foods; Salmonella; cocoa powder; process validation; oxidative quality.*

1 Introduction

Cocoa powder manufacture generally includes roasting or another upstream microbial reduction step, liquor production, hydraulic pressing, cake breaking, final milling and packaging. Cocoa cake formed after pressing is an intermediate solid whose porosity, fat distribution, water activity and mechanical history vary across lots and spatial positions. Industrial mapping has demonstrated radial fat heterogeneity in pressed cocoa cakes [1]. For a reactive gas applied to such a material, surface concentration cannot be presumed equivalent to exposure at the cake core.

The objective of this paper is to examine whether ozone, administered to size-controlled fragments before micronization, could provide a microbiologically useful treatment without altering the defining chemical and sensory properties of natural cocoa. This is an engineering hypothesis, not an assertion that ozone is superior to established thermal interventions.

2 Cocoa-cake structure and microbial hazard

2.1 Processing history and physical heterogeneity

The pressed solid contains nonfat cocoa components and residual butter in a mechanically consolidated network. Press temperature, pressure profile, liquor properties, cooling and fragmentation determine the geometry of accessible passages. A porous microstructure is not necessarily a gas-permeable microstructure: closed pores, fat-filled voids and diffusion-limited regions can restrict delivery. A physically coherent investigation must characterize fragment size, bulk and skeletal density, residual fat distribution, moisture, water activity and any accessible pore structure.

2.2 Microbiological relevance

Low water activity inhibits growth of many microorganisms but does not necessarily eliminate them. In particular, *Salmonella* may persist for prolonged periods in dry ingredients and may exhibit enhanced resistance to certain stresses [2,3]. Enterobacteriaceae and total aerobic counts are useful process indicators, but neither is a reliable surrogate for pathogen lethality. Bacterial spores and fungal propagules pose different susceptibility questions. Fungal inactivation also does not establish degradation or detoxification of pre-existing mycotoxins.

3 State of experimental evidence

A 2026 study by Khattra and colleagues [4] applied approximately 900–930 ppm gaseous ozone at 90% relative humidity for up to five hours to four artificially inoculated low-moisture foods, reporting strongly matrix-dependent *Salmonella* reductions. The results offer an important warning against transfer of treatment recipes between ingredients.

Matrix examined in [4]	Reduction after 5 h
Dried basil	5.0 ± 0.6 log CFU/g
Black peppercorns	2.4 ± 0.2 log CFU/g
Walnuts	1.7 ± 0.3 log CFU/g
Chia seeds	1.1 ± 0.0 log CFU/g

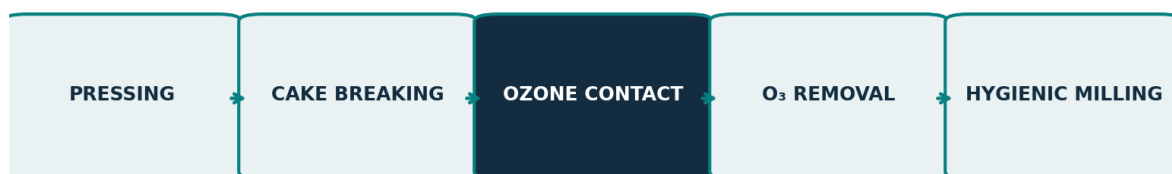
These values are published findings for the listed matrices and study conditions, not estimates for cocoa cake. The same study found *Enterococcus faecium* NRRL B-2354 to be a suitable surrogate only in its dried-basil system. A later 2026 study further examined the influence of relative humidity on basil [5]. Thus, a surrogate

accepted for heat treatment, or even for ozone on a different food, must not be assumed suitable for cocoa cake.

The directly cocoa-relevant physical literature documents cake-fat heterogeneity [1], whereas sufficiently comprehensive published cocoa-press-cake ozone challenge data remain unestablished in the cited evidence. This distinction is essential to the scientific claims of the present article.

4 Why fragmentation is the principal process variable

For an ideal cube of side length L , exposed geometric surface-to-volume ratio is $A/V = 6/L$. Breaking a cake into smaller fragments increases available external area and decreases the maximum distance from an interior location to an exposed surface. Neither change guarantees proportional microbial inactivation. The fragment-size distribution also influences entrainment, cohesion, pressure drop, attrition, explosion-risk management and post-treatment handling. Consequently, the optimum may occur at an intermediate fragment size rather than at the finest attainable powder.



Critical controls: fragment size · delivered exposure · moisture · oxidant demand · recontamination

Figure 1. Proposed process location and major control points. Conceptual engineering diagram; not an experimentally qualified process.

5 Transport, chemical demand and local ozone exposure

Ozone readily reacts with oxidizable constituents of biological and food matrices. Gas entering an industrial reactor can be depleted through wall losses, matrix reactions and gas-phase decomposition before penetrating an individual fragment. Therefore the concentration measured at the generator, inlet or chamber is not necessarily the concentration experienced at microbial locations.

A preliminary effective-medium model for a representative porous domain is $\partial C/\partial t = \nabla \cdot (D_{\text{eff}} \nabla C) - k_{\text{app}} C$, where C is local ozone concentration, D_{eff} an effective transport coefficient and k_{app} an apparent reaction-consumption constant. This is an illustrative hypothesis; convective pore transport, non-first-order reactions, variable humidity and evolving boundary conditions may demand a more complex model. The dimensionless grouping $\phi = L_c \sqrt{(k_{\text{app}}/D_{\text{eff}})}$, analogous to a Thiele modulus, suggests when consumption might dominate internal penetration.

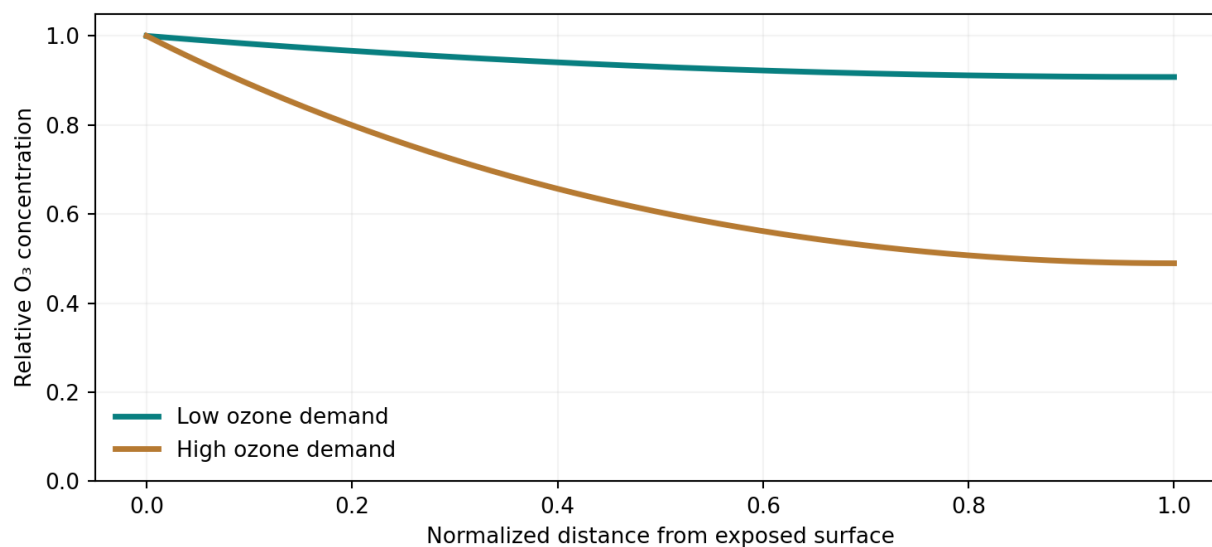


Figure 2. Illustrative normalized concentration profiles generated from a simplified reaction–diffusion solution. They are theoretical shapes, not measured ozone penetration into cocoa press cake.

The local integrated exposure $E(x) = \int_0^t C(x, \tau) d\tau$ provides a more defensible mechanistic descriptor than nominal concentration multiplied by clock time. Actual microbial lethality must still be fitted experimentally as a function of exposure, organism, water activity, temperature and matrix chemistry. No universal exposure-to-log-reduction coefficient is proposed.

6 Ozone-related effects on cocoa quality

An acceptable process must preserve the identity and marketability of natural, non-alkalized cocoa. Ozone may oxidize susceptible unsaturated moieties in residual cocoa butter and modify certain phenolics or volatile aroma constituents. The precise magnitude of these transformations under cake-specific conditions is unknown and must be determined, particularly for higher-fat cake grades.

Quality domain	Recommended endpoint
Lipid oxidation	Peroxide value, suitable secondary oxidation markers and selected volatiles
Color	CIELAB L*, a*, b* and ΔE relative to matched controls
Phenolic profile	Targeted HPLC of catechin, epicatechin and relevant flavanols
Aroma	HS-SPME–GC–MS with trained sensory testing
Physical behavior	Moisture, water activity, flowability, granulometry, dispersibility
Storage stability	Time-resolved testing against matched untreated controls

Changes must be assessed both immediately after processing and during storage. An absence of short-term changes in total phenolics, for example, does not rule out alteration of individual phenolic species. Likewise, a reduction in culture counts cannot justify an assertion that a chemical contaminant has been removed or rendered safe.

7 Proposed experimental validation program

7.1 Sampling, comparisons and controls

Collect multiple independent industrial lots with documented origin, pressing history and fat content. Establish baseline microbiology, water activity, moisture, oxidation markers, color and aroma. Randomize test units within lots, while respecting the hierarchical structure of independent batches, fragments and analytical replicates. Compare untreated compact cake, untreated fragmented cake, treated compact cake, treated coarse fragments, treated finer non-micronized fragments and -where relevant- treated finished powder.

7.2 Screening and dose measurement

A staged experimental design should screen characteristic fragment size, ozone inlet concentration, contact duration, relative humidity, product temperature, flow and bed loading. Measure inlet and outlet ozone concentration over time and document losses, rather than inferring the applied dose solely from generator nameplate capacity. Record residence-time distribution and check for reactor bypass or channeling. Screening should be followed by response-surface optimization only where initial data support identifiable main effects and interactions.

7.3 Spatial sampling and microbial recovery

Divide representative fragments into pre-specified external, intermediate and central regions and separately quantify recovered viable organisms. Spatial preparation procedures must avoid cross-contamination. Confirmatory experiments should measure the microbiological status of powder obtained by subsequent hygienic milling. The minimum reliably achieved regional log reduction, not merely the mean surface reduction, should inform safety qualification.

Any Salmonella challenge requires appropriate laboratory containment and expertise; an industrial surrogate must be validated specifically against the organism, matrix and ozonation conditions. Recovery media must account for potentially sublethally injured cells. Quantification below the analytical detection limit is censored information, not proof of sterility.

7.4 Acceptance criteria and uncertainty

Predefine the targeted pathogen reduction from the food-safety hazard analysis and downstream intended use. Separately establish customer-relevant thresholds for sensory difference, lipid oxidation, color, moisture and processability. Estimate effect sizes and confidence intervals across independent lots. A feasible operating window requires simultaneous satisfaction of microbial lethality, chemical quality, occupational safety and economic criteria; failing any critical endpoint invalidates the proposed operating point.

8 Proposed spatially resolved framework

Let $R(x)$ be a local log reduction and $E(x)$ the local integrated exposure. An empirical response $R(x) = f[E(x), a_w, T, \text{fat fraction, microorganism}]$ can be calibrated only after controlled challenge experiments. A quality-constrained feasible region may be written $\Omega = \{z : \min_x R(x,z) \geq R_{\text{target}} \text{ and } Q_i(z) \leq Q_{i,\text{limit}} \text{ for each critical quality endpoint } i\}$, where z contains the equipment operating variables. The optimization is conceptually original in combining minimum spatial lethality with multiple quality constraints; it is not a validated regulatory assessment method.

This framework encourages three complementary datasets: (i) reactor-scale mass balance and distribution, (ii) fragment-scale spatial survival, and (iii) finished-powder quality and microbiology. Agreement between these datasets is more informative than a favorable surface-count result in isolation.

9 Industrial engineering and occupational controls

Reactor options include enclosed agitated beds, moving beds and, where powder behavior permits, fluidized systems. Selection should follow particle-flow characterization, residence-time analysis and cleaning validation. Ozone exposure requires monitored containment, suitable construction materials, exhaust destruction, access interlocks and worker protection. Fragmentation and milling may create combustible organic dust; an ATEX assessment and ignition-source controls are necessary where applicable. Oxygen-enriched feed atmospheres require additional review. Hygienic separation after treatment is essential because downstream milling and packaging can reintroduce contamination.

10 European regulatory assessment

Compliance with Regulations (EC) No 178/2002 and 852/2004 is foundational but does not itself establish authorization to apply ozone directly to cocoa. The relevant distinction between processing-aid use, biocidal disinfection and other technological functions must be resolved for the intended operation. In Spain, the consolidated Royal Decree 773/2023 on processing aids [9] warrants a use-specific assessment; processing-aid rules are not fully harmonized across the European Union. The fact that ozone decomposes does not by itself establish either lawful use or toxicological absence of oxidation by-products.

Regulation (EU) 2023/915 imposes relevant maximum levels for specified contaminants. Ozone should not be presented as a substitute for upstream control of ochratoxin A, cadmium or other contaminants. Organic cocoa requires separate scrutiny under Regulation (EU) 2018/848 and applicable implementing legislation. Regulatory clearance and industrial validation should precede commercial treatment claims.

11 Industrial feasibility and alternatives

A business case must include generator and oxygen-supply costs, gas monitoring, ozone destruction, handling and fragmentation, reactor volume, throughput and hold-up time, analytical qualification, product losses and maintenance. The competing baseline should be a properly validated industrial process such as steam-based or other appropriate thermal treatment, with the same microbial target and product-quality requirements. Gaseous ozone should not be awarded presumed advantages in energy use, quality preservation or cost until equivalent industrial data exist.

12 Limitations, conclusions and research priorities

The evidence for gaseous ozone in low-moisture foods is real but strongly matrix-dependent. Direct, adequately controlled and replicable evidence for full-depth microbial lethality in natural cocoa press cake remains insufficient for prescribing a standard process. The key hypothesis is that treatment of controlled fragments between pressing and milling may improve gas access while avoiding some disadvantages of fine-powder processing. This hypothesis requires spatially resolved challenge studies, reactor-scale ozone mass balances and simultaneous oxidative-quality assessment.

The central research priority is to determine whether an operating region exists where minimum local pathogen reduction reaches a predefined target and natural cocoa quality remains commercially acceptable. Without such proof, the technology should be regarded as investigational, not as an established cocoa-cake sanitization step.

References

- [1] Melo LJ, Orjuela A, Guerrero E. Improvement of industrial processes in Colombia. Case study: Cocoa pressing process in a chocolate factory. *Chem Eng Res Des.* 2022;183:307–317. doi:10.1016/j.cherd.2022.05.013.
- [2] Beuchat LR, et al. Low-water activity foods: increased concern as vehicles of foodborne pathogens. *J Food Prot.* 2013;76:150–172. doi:10.4315/0362-028X.JFP-12-211.
- [3] Methods for the Control of Foodborne Pathogens in Low-Moisture Foods. *Annu Rev Food Sci Technol.* 2018. doi:10.1146/annurev-food-030117-012304.
- [4] Khattra AK, Wason S, Gowda NAN, Lafontaine S, Subbiah J, Acuff JC. Impacts of gaseous ozone and mild heating treatments on the inactivation of *Salmonella enterica* and *Enterococcus faecium* NRRL B-2354 and on the quality of low-moisture food products. *J Food Prot.* 2026;89(5):100750. doi:10.1016/j.jfp.2026.100750.
- [5] Khattra AK, et al. Increasing relative humidity enhances gaseous ozone efficacy for inactivation of *Salmonella enterica* and *Enterococcus faecium* NRRL B-2354 on dried basil leaves. *LWT.* 2026;253:119717. doi:10.1016/j.lwt.2026.119717.
- [6] Regulation (EC) No 178/2002 of the European Parliament and of the Council (General Food Law). EUR-Lex: <https://eur-lex.europa.eu/eli/reg/2002/178/oj>
- [7] Regulation (EC) No 852/2004 on the hygiene of foodstuffs. EUR-Lex: <https://eur-lex.europa.eu/eli/reg/2004/852/oj>
- [8] Commission Regulation (EU) 2023/915 on maximum levels for certain contaminants in food. EUR-Lex: <https://eur-lex.europa.eu/eli/reg/2023/915/oj>
- [9] Spain. Real Decreto 773/2023, de 3 de octubre, por el que se regulan los coadyuvantes tecnológicos utilizados en los procesos de elaboración y obtención de alimentos (consolidated text). <https://www.boe.es/buscar/act.php?id=BOE-A-2023-20563>
- [10] Regulation (EU) 2018/848 on organic production and labelling of organic products. <https://eur-lex.europa.eu/eli/reg/2018/848/oj>
- [11] Directive 2014/34/EU on equipment and protective systems intended for use in potentially explosive atmospheres. <https://eur-lex.europa.eu/eli/dir/2014/34/oj>

Publication statement

Article category: critical scientific–technical review and methodological proposal. Figures are original conceptual.