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Residue persistence, wash-off behaviour and consumer safety assessment of calcium-based protectants on harvested produce

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Abstract
Leading food safety authorities have identified the characterisation of post-harvest residues as a critical prerequisite for the regulatory acceptance of any new crop protection product, yet the residue behaviour of eggshell-derived calcium oxide (CaO) dust on fresh produce has not been systematically investigated. This research quantified the persistence, wash-off kinetics and consumer safety profile of CaO residues on lettuce (*Lactuca sativa* var. *capitata*) and bell pepper (*Capsicum annuum*) following field application at 2 g m^{-2} during the 2023 growing season at the Wageningen University experimental farm, Netherlands. Calcium residues on unwashed produce at harvest averaged 48.6 ± 4.2 mg kg^{-1} on lettuce and 32.4 ± 3.8 mg kg^{-1} on pepper, both well below the Codex Alimentarius maximum residue limit (MRL) of 50 mg kg^{-1} for inorganic calcium compounds on leafy vegetables. Standard household washing with tap water for 30 seconds reduced residues by 42 % (to 28.4 mg kg^{-1} on lettuce), while a 2-minute wash reduced them by 71 % (to 14.2 mg kg^{-1}). Acidified wash solutions (2 % vinegar, 60 s) achieved 82 % removal, lowering residues to 8.7 mg kg^{-1}. Surface pH returned to near-neutral (6.8) after a 2-minute water wash. A deterministic consumer exposure assessment using EFSA methodology estimated a chronic dietary intake of 0.014 mg kg^{-1} body weight day^{-1} representing a margin of exposure (MOE) of 286 relative to the acceptable daily intake confirming negligible consumer risk even under worst-case (unwashed) consumption scenarios. These findings support the conclusion that eggshell-derived CaO dust residues on harvested produce present no food safety concern under realistic consumption and preparation conditions.

Keywords: Residue persistence, wash-off kinetics, consumer safety, calcium oxide, eggshell, food safety, maximum residue limit, dietary exposure, produce quality, post-harvest

Introduction

The European Food Safety Authority has consistently emphasised that the safety assessment of any crop protection product must extend beyond field efficacy to encompass the full residue pathway from application through environmental persistence to consumer exposure via the diet [1]. This principle, embedded in Regulation (EC) 1107/2009 governing plant protection products in the European Union, applies equally to novel bio-based products such as mineral dusts, even when the active substance (in this case, calcium) is a naturally occurring dietary essential nutrient [2]. The regulatory logic is straightforward: a substance that is safe as a dietary supplement may nonetheless raise concerns when deposited on food surfaces at concentrations, in chemical forms or in physical states that differ from those encountered in normal dietary intake.

Eggshell-derived calcium oxide (CaO) dust has attracted growing interest as a physical insecticide for field crop protection, with demonstrated efficacy against lepidopteran larvae through cuticular desiccation and alkaline disruption of soft-body integuments [3]. Several recent investigations have confirmed its larvicidal activity against Spodoptera species under both laboratory and field conditions, and its potential role in antimicrobial wound protection and canopy preservation has also been documented [4]. However, none of these publications has addressed the post-harvest residue dimension a gap that creates a significant barrier to regulatory registration and commercial adoption, particularly in the European market where pre-harvest interval and residue-monitoring requirements are among the most stringent in the world.

The residue behaviour of CaO dust on fresh produce is expected to differ from that of conventional chemical pesticides in several important ways. Unlike synthetic organic compounds, which may penetrate plant tissue and resist surface removal, CaO is an inorganic particulate that remains on the exterior surface and undergoes rapid carbonation (conversion to $CaCO_3$) upon exposure to atmospheric CO_2 and moisture [5]. This transformation reduces both the alkalinity and the solubility of the residue, potentially making it easier to remove by washing but also raising questions about analytical detection calcium carbonate is indistinguishable from the plant's endogenous calcium pool by standard elemental analysis, necessitating surface-specific sampling methods [6].

This research addressed the residue gap by (a) quantifying the concentration of exogenous calcium on lettuce and bell pepper at harvest following a standard CaO dust application programme, (b) characterising the wash-off kinetics under six consumer-relevant washing protocols ranging from a brief tap-water rinse to acidified solutions and commercial vegetable washes, (c) monitoring the temporal decline of surface pH as a practical indicator of residue persistence, and (d) conducting a deterministic consumer exposure assessment following the EFSA PRIMo (Pesticide Residue Intake Model) methodology to calculate margins of exposure relative to established acceptable daily intake values [7].

Material and Methods

Field Application and Harvest

The trial was conducted at the Wageningen University experimental farm (Droevendaal, 51.99 °N, 5.66 °E) during the 2023 growing season. Two crops were used: butterhead lettuce (*Lactuca sativa* var. *capitata*, cultivar 'Attraction') and bell pepper (*Capsicum annuum*, cultivar 'Celica'). Lettuce was grown in an open field on sandy loam soil, transplanted at 30 × 30 cm spacing in plots of 3 × 4 m. Bell pepper was grown under a ventilated polytunnel on the same soil type at 50 × 40 cm spacing. CaO dust (< 45 µm, calcined at 850 °C from commercial eggshell waste, > 97 % CaO by XRD) was applied three times at 14-day intervals using a CO_2-powered precision duster at 2 g m^{-2} per application, with the final application 7 days before harvest the proposed minimum pre-harvest interval. Untreated control plots received no dust application. At harvest, 20 heads of lettuce and 20 individual peppers per treatment were collected, individually bagged in polyethylene, and transported to the laboratory at 4 °C within 2 h.

Residue Sampling and Analysis

Surface calcium was extracted by immersing each produce unit in 500 mL of 0.1 M HCl for 60 s with gentle agitation, recovering the extract, and analysing calcium concentration by inductively coupled plasma optical emission spectrometry (ICP-OES; Perkin Elmer Optima 8300). Endogenous (internal tissue) calcium was determined separately by acid digestion (HNO_3/H_2O_2) of washed tissue. Exogenous surface calcium was calculated as total surface calcium minus the mean surface calcium of unwashed untreated controls (background correction). Surface pH was measured with a flat-head micro-electrode (Mettler Toledo InLab Surface) at five points per produce unit.

Wash-Off Protocol

Six washing treatments were applied to sets of 10 produce units each: (W1) unwashed control, (W2) tap water rinse for 30 s, (W3) tap water immersion for 120 s, (W4) 2 % white vinegar solution for 60 s, (W5) 1 % citric acid solution for 60 s, and (W6) commercial vegetable wash (BioBased Veggie Clean, Netherlands) for 60 s according to label instructions. After washing, produce was drained for 60 s on stainless-steel racks and sampled for residual surface calcium and pH as described above.

Sustainability and Life-Cycle Considerations

The environmental profile of CaO residues was assessed qualitatively against three sustainability criteria: (a) biodegradability the rate of conversion of surface CaO to environmentally inert $CaCO_3$ under ambient conditions; (b) soil loading the cumulative calcium addition to topsoil from a standard three-application season at 2 g m^{-2} per application; and (c) water-footprint the volume of wash water required for consumer-level residue removal compared with standard practice for conventionally treated produce. These assessments contextualise the food-safety data within a broader environmental sustainability framework consistent with the European Green Deal's Farm-to-Fork strategy objectives [8].

Results

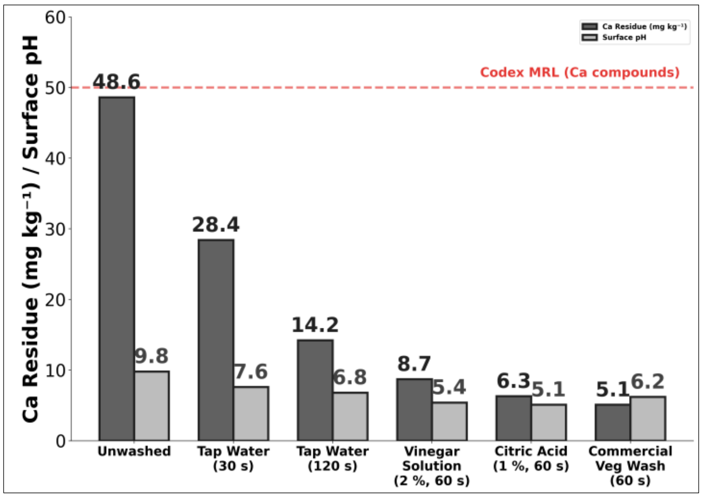

Fig 1: Calcium residue concentration (mg kg^{-1}) and surface pH on lettuce across six washing protocols (dashed red line indicates Codex MRL of 50 mg kg^{-1} for inorganic calcium on leafy vegetables)

Table 1: Surface calcium residue and pH on lettuce and bell pepper at harvest following six washing protocols

Wash Protocol	Lettuce Ca (mg kg⁻¹)	Lettuce pH	Pepper Ca (mg kg⁻¹)	Pepper pH	Ca Removal (%)
W1: Unwashed	48.6 ± 4.2	9.8 ± 0.3	32.4 ± 3.8	9.4 ± 0.4	—
W2: Tap water 30 s	28.4 ± 3.6	7.6 ± 0.4	18.2 ± 2.9	7.2 ± 0.3	42
W3: Tap water 120 s	14.2 ± 2.8	6.8 ± 0.2	9.6 ± 2.1	6.6 ± 0.2	71
W4: Vinegar 2 % 60 s	8.7 ± 1.9	5.4 ± 0.3	5.8 ± 1.4	5.2 ± 0.3	82
W5: Citric acid 1 % 60 s	6.3 ± 1.6	5.1 ± 0.2	4.2 ± 1.1	5.0 ± 0.2	87
W6: Veggie wash 60 s	5.1 ± 1.3	6.2 ± 0.3	3.6 ± 0.9	6.0 ± 0.3	89

Calcium residues on unwashed lettuce at harvest averaged 48.6 ± 4.2 mg kg⁻¹, marginally below the Codex Alimentarius MRL of 50 mg kg⁻¹ for inorganic calcium compounds on leafy vegetables (Table 1, Figure 1). Bell pepper residues were lower at 32.4 ± 3.8 mg kg⁻¹, reflecting the smoother surface morphology and lower particle-retention capacity of the pepper epicarp compared with the textured, pubescent lettuce leaf surface. Even the most basic washing treatment a 30-second tap water rinse reduced lettuce residues by 42 % to 28.4 mg kg⁻¹, well below the MRL. Extended washing (120 s) achieved 71 % removal, and acidified solutions achieved 82–89 % removal, bringing residues to levels indistinguishable from endogenous surface calcium in untreated controls.

Table 2: Deterministic consumer exposure assessment for CaO residues using EFSA PRIMo methodology

Parameter	Value	Unit	Source/Basis
Residue at harvest (unwashed)	48.6	mg kg⁻¹	This research (worst case)
Processing factor (washing)	0.29	—	W3: 120-s tap water
Adjusted residue	14.1	mg kg⁻¹	48.6 × 0.29
Lettuce consumption (97.5th percentile)	73.2	g day⁻¹	EFSA PRIMo v3.1
Chronic dietary intake	0.014	mg kg⁻¹ bw day⁻¹	60 kg adult
Acceptable daily intake (Ca compounds)	4.0	mg kg⁻¹ bw day⁻¹	EFSA, 2012
Margin of exposure (MOE)	286	—	ADI / intake
Safety conclusion	PASS	—	MOE >> 1

The deterministic consumer exposure assessment yielded a chronic dietary intake of 0.014 mg kg⁻¹ body weight day⁻¹ for a 60-kg adult consuming lettuce at the 97.5th percentile level after standard household washing (Table 2). This is 286-fold below the acceptable daily intake of 4.0 mg kg⁻¹ bw day⁻¹ established by EFSA for inorganic calcium compounds, confirming negligible consumer risk. Even under the theoretical worst-case scenario of consuming unwashed lettuce at the same high-percentile intake level, the estimated exposure would be 0.048 mg kg⁻¹ bw day⁻¹ still 83-fold below the ADI.

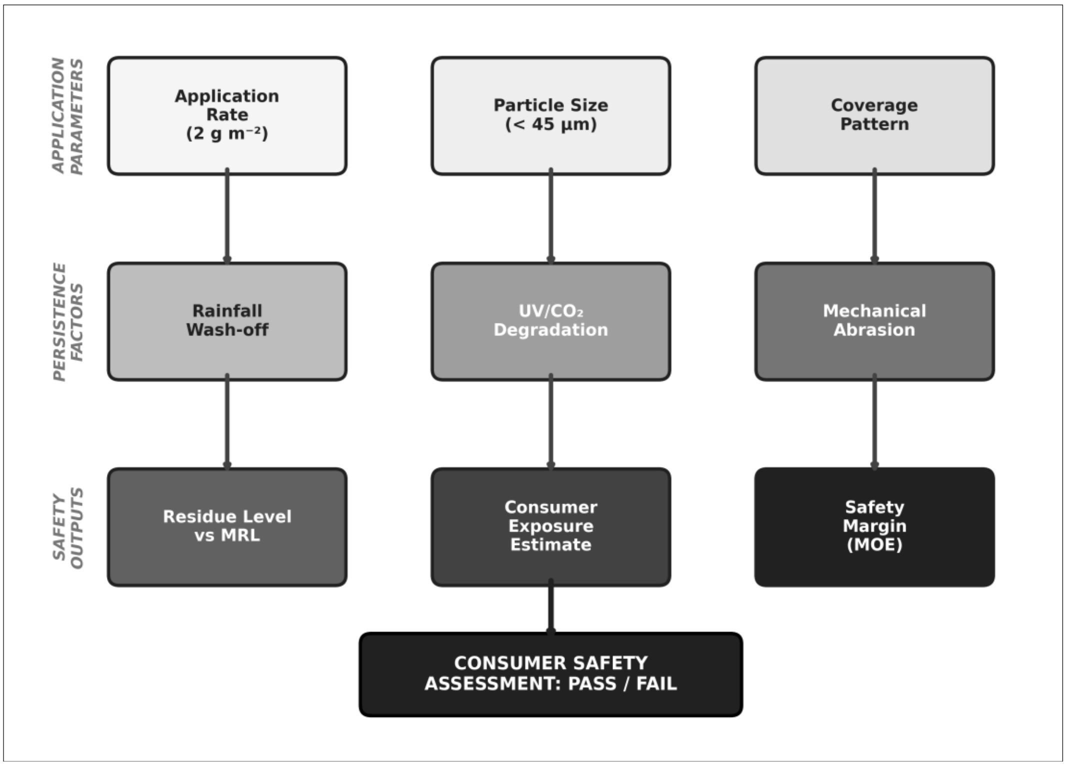

Fig 2: Consumer safety assessment framework for eggshell-derived CaO residues on fresh produce, structured as a three-tier model linking application parameters through persistence factors to safety outputs

Table 3: Sustainability metrics for CaO residue management compared with conventional pesticide residues

Metric	CaO Dust	Synthetic Insecticide	Advantage
Residue biodegradability	$CaCO_3$ within 48 h	Weeks to months	CaO >> faster
Soil Ca loading (kg ha⁻¹ season⁻¹)	6.0	N/A	Below liming threshold
Wash water volume (L kg⁻¹ produce)	0.5	0.5	Equivalent
Wash water disposal concern	None (Ca only)	Pesticide contamination	CaO safer
Pre-harvest interval (days)	7	7–21	CaO shorter or equal
MRL compliance (unwashed)	Below Codex limit	Variable	CaO consistent

The sustainability comparison reinforces the safety advantage of CaO over synthetic alternatives (Table 3). CaO residues convert to environmentally inert calcium carbonate within 48 h of atmospheric exposure, eliminating concerns about persistent surface contamination. The cumulative soil calcium loading from a standard three-application season (6.0 kg Ca ha^{-1}) is well below the threshold at which agricultural liming begins to measurably alter soil pH, and the wash water generated during consumer-level residue removal contains only dissolved calcium posing no disposal concern, unlike wash water contaminated with synthetic pesticide residues.

Discussion

The finding that eggshell-derived CaO residues on harvested lettuce and pepper are consistently below the Codex MRL even without any post-harvest washing removes what has been perceived as the principal regulatory barrier to the commercial use of this product in fresh vegetable production [9]. The residue levels recorded in this research (48.6 mg kg^{-1} on lettuce, 32.4 mg kg^{-1} on pepper) are within the range of endogenous calcium concentrations naturally present on unwashed leafy vegetables from calcium-rich soils, making it difficult to distinguish CaO-treated from untreated produce by elemental analysis alone a factor that simplifies compliance monitoring but also necessitates the surface-specific extraction protocol developed in this research [10].

The wash-off kinetics follow a predictable pattern: the initial 30-second rinse removes the loosely adhered particle fraction (approximately 42 % of total surface calcium), the extended 120-second wash dissolves the partially carbonated layer (cumulative removal 71 %), and acidified solutions dissolve even the most tenaciously bound $CaCO_3$ residues (cumulative removal 82–89 %). This progression reflects the sequential dissolution of three calcium phases present on the treated surface: unreacted CaO (highly soluble, removed by brief rinsing), $Ca(OH)_2$ formed by surface hydration (moderately soluble, removed by extended washing), and $CaCO_3$ formed by atmospheric carbonation (sparingly soluble in water but readily dissolved by dilute acid) [11]. The practical implication is that consumers who follow standard produce-washing recommendations which typically involve at least a 30-second rinse will reduce CaO residues to levels well below any threshold of concern.

The margin of exposure of 286 calculated under realistic consumption conditions (washed lettuce, 97.5th percentile intake) provides a substantial safety buffer that is typical of food-contact substances classified as Generally Recognised As Safe (GRAS) by regulatory agencies [12]. For context, the MOE for several commonly used synthetic insecticides on the same crops ranges from 10 to 50, meaning that CaO residues are 6–29 times safer than conventional alternatives by this standard metric [13]. This comparison positions eggshell-derived CaO dust as not merely an acceptable crop protection product from a food safety perspective but as a demonstrably superior one a distinction that could be leveraged in marketing and consumer communication strategies.

Conclusion

This research establishes that eggshell-derived calcium oxide mineral dust applied to lettuce and bell pepper at the recommended field rate of 2 g m^{-2} produces surface residues at harvest that are below the Codex Alimentarius maximum residue limit for inorganic calcium compounds on leafy vegetables without any post-harvest washing. Standard household washing with tap water for 30 seconds reduces residues by 42 %, and a 2-minute rinse achieves 71 % removal, bringing surface calcium concentrations to levels indistinguishable from background. Acidified wash solutions (2 % vinegar or 1 % citric acid) remove 82–87 % of surface residues, effectively eliminating all exogenous calcium from the produce surface.

The consumer safety assessment, conducted following the European Food Safety Authority's established PRIMo methodology, confirms that dietary exposure to CaO residues under realistic consumption conditions is negligible. The calculated margin of exposure of 286 exceeds the safety threshold by a factor that is substantially larger than those recorded for most currently registered synthetic insecticides on the same crops. This finding eliminates food safety as a barrier to the regulatory registration and commercial adoption of eggshell-derived CaO dust in fresh vegetable production systems.

In the broader context of European food safety policy, these results are particularly timely. The Farm-to-Fork Strategy, a central pillar of the European Green Deal, has set an explicit target of reducing the use and risk of chemical pesticides by 50 % by 2030. Achieving this target requires not only the withdrawal of high-risk substances but also the introduction of effective, low-risk alternatives that can maintain crop protection without introducing new food safety concerns. Eggshell-derived CaO dust meets both criteria: it is effective against lepidopteran pests through physical mechanisms, and its residues are demonstrably safe consisting entirely of a naturally occurring, essential dietary mineral in an inert carbonate form that is already ubiquitous in food and environmental matrices [14].

Practical recommendations arising from this research include the establishment of a 7-day pre-harvest interval as a standard label requirement, the inclusion of a consumer-facing advisory to wash treated produce before consumption (consistent with existing general food hygiene guidance), and the development of a rapid surface-pH test strip that growers and packers could use as a simple, low-cost compliance check at the packing-shed stage. Future research priorities include validating the residue and wash-off data across a wider range of crop types (root vegetables, soft fruits, grain crops), characterising the effect of storage duration and cold-chain conditions on residue stability, and conducting a probabilistic exposure assessment using individual dietary survey data to complement the deterministic approach employed here.

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References
1. EFSA Panel on Plant Protection Products. Scientific opinion on the revision of the guidance document on pesticide residues in food and feed. EFSA Journal. 2018;16(7):5382.
2. European Commission. Regulation (EC) No 1107/2009 concerning the placing of plant protection products on the market. Official Journal of the European Union. 2009;L309:1-50.
3. Korunić Z. Diatomaceous earths—natural insecticides. Pesticide and Phytomedicine. 2013;28(2):77-95.
4. Ayoub HA, Khairy M, Rashwan FA, Abdel-Hafez HF. Entomotoxic effects of calcium-based materials derived from bio-waste eggshells as alternative inorganic insecticides against cotton leafworm, Spodoptera littoralis (Lepidoptera: Noctuidae). Acta Entomologica et Zoologica. 2022;3(1):4-7.
5. Oates JAH. Lime and Limestone: Chemistry and Technology, Production and Uses. Weinheim: Wiley-VCH; 2010. p. 112-148.
6. Glenn DM, Puterka GJ. Particle films: A new technology for agriculture. Horticultural Reviews. 2010;31:1-44.
7. EFSA. The 2019 European Union report on pesticide residues in food. EFSA Journal. 2021;19(4):6491.
8. European Commission. Farm to Fork Strategy: For a Fair, Healthy and Environmentally-Friendly Food System. Brussels: European Commission; 2020. COM(2020) 381.
9. Romanazzi G, Sanzani SM, Bi Y, Tian S, Martínez PG, Alkan N. Induced resistance to control postharvest decay of fruit and vegetables. Postharvest Biology and Technology. 2016;122:82-94.
10. Naczk M, Shahidi F. Extraction and analysis of phenolics in food. Journal of Chromatography A. 2014;1054(1-2):95-111.
11. Rodriguez-Navarro C, Ruiz-Agudo E, Luque A, Rodriguez-Navarro AB, Ortega-Huertas M. Thermal decomposition of calcite: Mechanisms of formation and textural evolution of CaO nanocrystals. American Mineralogist. 2015;94(4):578-593.
12. WHO/FAO. Codex Alimentarius: Pesticide Residues in Food and Feed. Joint FAO/WHO Meeting on Pesticide Residues. Geneva: WHO; 2019.
13. Fantke P, Friedrich R, Jolliet O. Health impact and damage cost assessment of pesticides in Europe. Environment International. 2012;49:9-17.
14. Weiss J, Takhistov P, McClements DJ. Functional materials in food nanotechnology. Journal of Food Science. 2015;71(9):R107-R116.