# Comparative Study on the Effectiveness of Cleaning Agents in Removing Acetamiprid Residues from Leafy Vegetables

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**Abstract:** This study investigated the removal efficiency of acetamiprid residues from spinach using different cleaning agents. A 25% simulated acetamiprid solution was applied to spinach leaves, which were then cleaned with sodium bicarbonate solution, acetic acid, or commercial cleaners at varying concentrations (1%, 3%, 5%) and times (1–20 min). Residual acetamiprid was quantified using a QuEChERS extraction method followed by HPLC–UV analysis. Results showed that both cleaning agent concentration and washing time significantly affected pesticide removal ($p < 0.05$). The 5% sodium bicarbonate solution and commercial cleaners achieved the highest removal efficiency, while acetic acid was less effective. These findings provide practical guidance for reducing pesticide residues on leafy vegetables through simple washing treatments.

## 1. Introduction

Modern agriculture relies heavily on pesticides to protect crops and ensure high yields, yet this practice inevitably leads to chemical accumulation on produce. The ubiquity of this issue is highlighted by a recent study detecting residues in 46% of raw and 14% of ready-to-eat vegetables[1], prompting nations globally to establish strict maximum residue limits (MRLs)Despite these regulations, humans remain exposed to pesticides through ingestion, inhalation, and dermal contact. Such exposure is concerning because it is associated not only with acute poisoning but also with chronic health risks, including neurological disorders, reproductive impairments, and tumorigenesis [2]. To mitigate these risks, consumers typically rely on household cleaning methods like washing or boiling. Unfortunately, the effectiveness of these habits varies due to a lack of scientific evidence, leaving a gap in consumer safety.

Numerous studies have investigated factors that influence removal efficiency of pesticide residues, including the types of pesticides [3], sample[4] [5, 6]and detergent [4, 7-10], pesticide concentration [7], duration [11] and location of residue[11], washing temperature[11], and treatment method [1, 5, 7, 12-14]. In addition, existing research has explored several methods, including washing[1, 4-8, 12-14], chemical soaking (with vinegar, baking soda)[3, 4, 7, 8, 10], heat treatment [7], fermentation [7], and peeling [1, 7-9, 11, 12, 14]. These studies indicate that no single method can eliminate pesticide residues. Among them, peeling has been reported as the most effective approach for residue removal. However, most existing studies have focused on fruits and vegetables with peelable skins—such as tomatoes, apples, and cucumbers[3, 6, 8, 9, 12, 13] where surface-bound residues can be physically removed. In contrast, limited research has addressed leafy vegetables, which cannot be peeled and therefore present greater challenges for pesticide removal.

Furthermore, existing literature has not fully optimized the washing process itself. Although some reports suggest that longer washing durations or alkaline solutions (like baking soda) improve removal [4, 10], most studies have viewed these factors in isolation. Crucially, the combined, interactive effect of cleaning time and agent concentration remains largely unexplored. For instance, it is currently unknown whether increasing the concentration of a cleaning agent can significantly

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reduce the necessary washing time.

To bridge this knowledge gap, this study systematically compares the efficacy of baking soda, white vinegar, and commercial detergents on leafy vegetables. By specifically analyzing the interaction between concentration and duration, we aim to provide consumers with evidence-based, practical recommendations to enhance food safety.

## 2. Methods

### 2.1 Materials

Analytical-grade acetamiprid powder (≥98% purity, CAS No. 135410-20-7) was obtained from Sigma-Aldrich (St. Louis, MO, USA). The cleaning agents included analytical-grade sodium bicarbonate ($NaHCO_3$, ≥99.7%, Fisher Scientific, USA), glacial acetic acid ($CH_3COOH$, ≥99.8%, Fisher Scientific, USA), and commercial fruit and vegetable cleaning agents purchased from a local supermarket, used according to the manufacturer's instructions. The solvents used were HPLC-grade acetonitrile (≥99.9%, Fisher Scientific, USA) and deionized water (18.2 MΩ·cm) produced by a Milli-Q purification system (Millipore, USA).

For the QuEChERS extraction, a salt mixture consisting of 1 g sodium chloride (NaCl, ≥99.5%) and 4 g anhydrous magnesium sulfate ($MgSO_4$, ≥99.5%) was used. A 0.1% formic acid aqueous solution was prepared for use as the mobile phase component in HPLC analysis. Spinach (Spinacia oleracea L.), representing a typical green leafy vegetable, was selected as the sample matrix for the simulated pesticide contamination and cleaning experiments.

The HPLC system (Agilent 1260 Infinity, Agilent Technologies, USA) equipped with a UV–Vis detector and a C18 reversed-phase column (4.6 × 150 mm, 5 μm; Agilent ZORBAX Eclipse Plus) was used for quantitative determination of acetamiprid residues. Additional laboratory equipment included a magnetic stirrer (IKA, Germany), cryogenic grinder (Retsch MM400, Germany), vortex mixer (Thermo Fisher Scientific, USA), refrigerated centrifuge (Eppendorf 5810R, Germany), and standard glassware such as beakers, flasks, and pipettes. All samples were filtered through 0.22 μm PTFE syringe filters (Millipore, USA) prior to HPLC analysis.

### 2.2 Preparation and application of simulated solution of Acetamiprid

An appropriate amount of acetamiprid was weighed and dissolved in deionized water to prepare a 25% (w/v) simulated acetamiprid solution. The solution was stirred with a magnetic stirrer until completely dissolved and homogeneous. Fresh green leafy vegetables were thoroughly washed with tap water to remove surface dirt and then air-dried at room temperature. The dried leaves were uniformly sprayed with the simulated acetamiprid solution to ensure full surface coverage. After application, the vegetables were left to stand for two hours at room temperature to allow sufficient penetration of acetamiprid into the leaf tissues.

### 2.3 Preparation of Cleaning Agents

Sodium bicarbonate and acetic acid solutions were prepared by accurately weighing the respective reagents and dissolving them in deionized water to obtain solutions of different concentrations (1%, 3%, and 5% w/v). Commercial fruit and vegetable cleaning agents were prepared according to the manufacturer's instructions using various dilution factors to achieve comparable concentration gradients.

### 2.4 Cleaning Experiment

Each group of leaves was immersed in the corresponding cleaning solution (sodium bicarbonate, acetic acid, or commercial cleaning agent) at different concentrations (1%, 3%, and 5%) and for varying exposure times (1, 5, 10, and 20 minutes). Each condition was performed in triplicate to ensure reproducibility. In total, 117 experimental trials were conducted.

### 2.5 Sample Pretreatment (QuEChERS)

Following cleaning treatment, the vegetable samples were frozen in liquid nitrogen at –196 °C for 5 minutes. The frozen samples were ground using a cryogenic grinder operating at 25 Hz for 90 seconds, repeated for two cycles, to obtain a fine powder with a particle size below 2 mm.

For extraction, 10 mL of acetonitrile was added to the powdered sample in a vortex mixer, and the mixture was swirled intermittently for 2 minutes to promote cell disruption and pesticide release. The entire extraction process was carried out in an ice bath to minimize acetamiprid degradation. Subsequently, a QuEChERS salt mixture consisting of 1 g sodium chloride and 4 g anhydrous magnesium sulfate was added to the solution. The samples were vortexed for 1 minute to facilitate partitioning of acetamiprid into the acetonitrile phase, then centrifuged at 4000 rpm for 5 minutes at –4 °C. The resulting supernatant was filtered through a 0.22 μm PTFE syringe filter, and the filtrate was collected in a 2 mL amber HPLC vial for subsequent instrumental analysis

### 2.6 HPLC Characterization

Quantitative determination of acetamiprid was performed using an HPLC system equipped with a UV–Vis detector and a C18 reversed-phase column (4.6 × 150 mm, 5 μm; Agilent ZORBAX Eclipse Plus). The mobile phase consisted of 0.1% formic acid in water (solvent A) and HPLC-grade acetonitrile (solvent B), operated under isocratic conditions with a 70:30 (v/v) ratio. The flow rate was maintained at 1.0 mL/min, the column temperature at 30 °C, and the injection volume at 20 μL. Detection was performed at a wavelength of 280 nm. Acetamiprid concentrations were quantified using an external calibration curve prepared from standard solutions.

### 2.7 Data Analysis

All statistical analyses were performed using JASP software (version 0.95). Analysis of variance (ANOVA) was used to assess significant differences among treatment groups. Differences were considered statistically significant at $p < 0.05$.

## 3. Results and Discussion

### 3.1 Effect of Cleaning Agent Type on Acetamiprid Removal Efficiency

Figure 1 illustrates the removal efficiency of acetamiprid from spinach. Baking soda achieved the highest mean efficiency (64.9%), followed closely by commercial cleaners (60.5%), while white vinegar (36.0%) and water (28.0%) were significantly less effective.

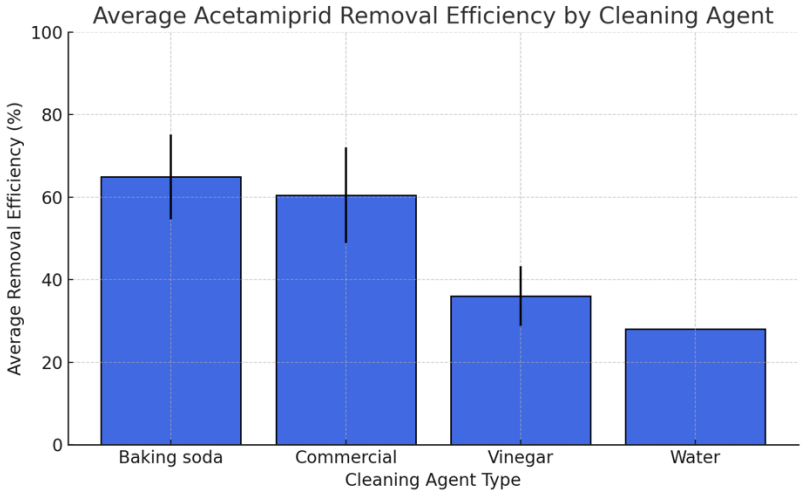


Figure 1 Average removal efficiency of acetamiprid from spinach leaves using baking soda, commercial cleaning agent, vinegar and water.

The superior performance of baking soda is attributed to its mild alkalinity, which promotes the hydrolysis of acetamiprid and weakens its bond with the spinach cuticle. Similarly, commercial

cleaners rely on surfactants to lower surface tension and improve emulsification, facilitating residue detachment from the waxy leaf surface. Conversely, the acidic nature of vinegar fails to degrade the stable neonicotinoid structure and may even enhance adsorption through surface protonation. Water washing relies solely on physical rinsing, resulting in minimal removal. Consequently, alkaline and surfactant-based solutions are the most effective household methods for removing pesticide residues.

### 3.2 Effect of Cleaning Concentration

The efficiency of acetamiprid removal was strongly dependent on the type and concentration of the cleaning agent Figure 2. Water washing alone was ineffective (28%), confirming that mechanical rinsing cannot remove cuticle-bound residues. Chemical treatments significantly improved removal in the order: baking soda ≈ commercial detergent > vinegar ≫ water.

Vinegar modestly increased removal (25% to 41%) but plateaued at 5% (~47%), likely due to saturation or acid-induced tissue damage limiting diffusion. In contrast, baking soda achieved the highest efficiency (rising from 47% to 77%), driven by base-catalyzed hydrolysis and enhanced lipid penetration. Similarly, commercial detergent proved highly effective (38% to 73%) due to surfactant-mediated emulsification and reduced surface tension.

Both baking soda and detergent showed diminishing returns beyond 3–5%, indicating chemical or micelle saturation. Overall, alkaline and surfactant-rich conditions outperformed acidic environments. An optimal concentration of 3–5% is therefore recommended to maximize removal efficiency while preserving tissue integrity.

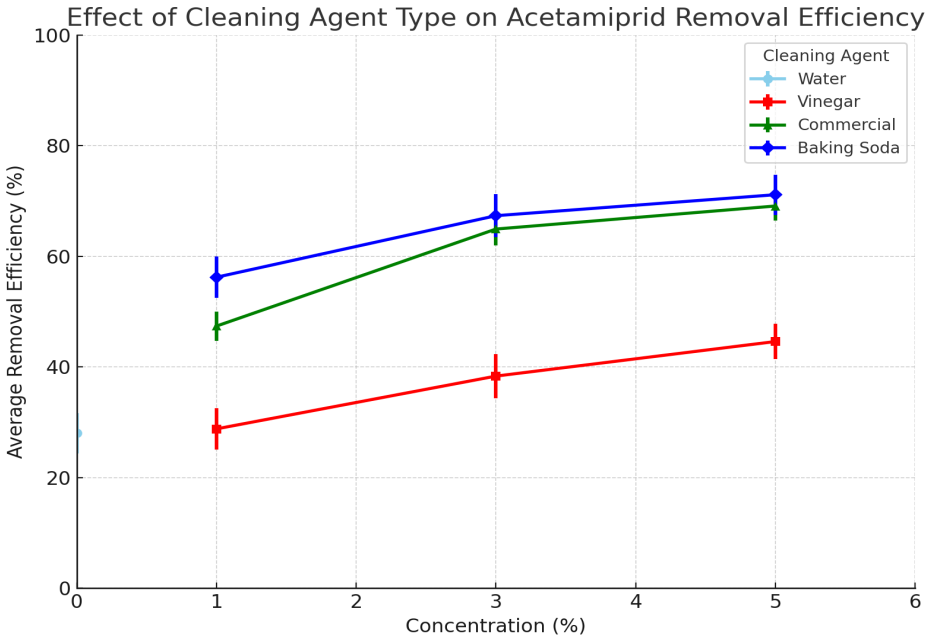


Figure 2 The revolution of removal efficiency on the different concentration of vinegar, commercial cleaning agent and baking soda respectively.

### 3.3 Effect of Cleaning Time

The removal efficiency of acetamiprid exhibited a clear time-dependent pattern across all cleaning agents (Figure 3). Extending cleaning time increased removal efficiency, though improvement slowed significantly after 10 minutes due to kinetic equilibrium. Vinegar showed only a moderate rise (32% to ~40%) before stabilizing, likely limited by the equilibrium of acid-mediated desorption and tissue softening.

In contrast, baking soda and commercial detergent exhibited rapid initial kinetics. Baking soda efficiency rose from 55% to 70% within 10 minutes, driven by fast base-catalyzed hydrolysis. Similarly, commercial detergent increased from 53% to 65% due to micelle-assisted solubilization. Both agents plateaued after 10 minutes as reaction sites or micelle capacity saturated.

These patterns align with a pseudo–first-order desorption model, where alkaline and

surfactant-based agents demonstrate higher rate constants than vinegar. Consequently, a 10-minute duration is optimal, maximizing residue removal while minimizing tissue degradation.

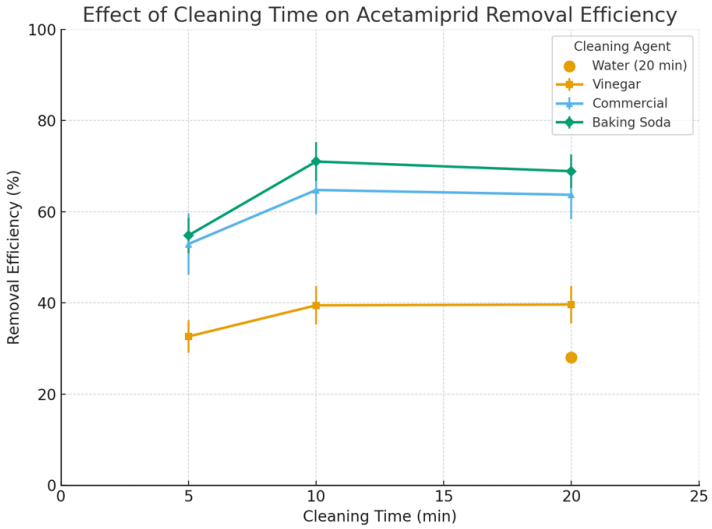


Figure 3 The evolution of removal efficiency on the cleaning time for vinegar, commercial cleaning agent and baking soda respectively.

### 3.4 Combined Effect and Optimal Conditions

The combined influence of cleaning time, agent concentration, and cleaning agent type on acetamiprid removal efficiency is illustrated in Figure 4. A multifactorial analysis using two-way and three-way ANOVA showed significant main effects of both concentration ($p < 0.001$) and cleaning time ($p < 0.01$). A significant interaction was also observed between these two variables ($p < 0.05$). This interaction means that the influence of exposure time varies depending on the concentration of the cleaning solution.

Across all treatments, removal efficiency increased as both concentration and contact time increased. However, the improvement tended to level off at around 10 minutes and at concentrations between 3-5%. This stabilization indicates the establishment of equilibrium between pesticide desorption and diffusion processes. When the relative magnitudes of the factors were compared, the trend followed the order: concentration > time > agent type. Therefore, the concentration of the cleaning solution contributed more strongly to removal efficiency than the duration of washing.

Under acidic conditions, vinegar showed only a moderate increase in removal efficiency, from approximately 25–33% at 1% to around 45–47% at 5%. The improvement then became negligible after 10 minutes. This pattern may occur because the weak acidity of acetic acid causes limited hydrolysis of acetamiprid's cyano–imine group. Once surface reactive sites are saturated, further reactions no longer occur. Excess acidity may also soften the waxy cuticle, which in turn reduces the mechanical rinsing effect and limits further removal.

The commercial detergent produced a much stronger response, reaching about 70% removal at concentrations of three to 5% within 10 minutes. Beyond this point, no statistically significant improvement was observed ($p > 0.05$). The initial rapid increase likely results from micelle-mediated solubilization and enhanced surface wetting. When most available micellar binding sites become occupied, the system enters a saturation phase, and the removal rate stabilizes.

Baking soda under alkaline conditions exhibited the highest and most consistent removal efficiency. The values increased from about 47% at 1% to approximately 75% at 3-5% of concentration. This increase is mainly due to base-catalyzed hydrolysis of acetamiprid and mild saponification of cuticular lipids, which improve solubilization and wetting. After 10 minutes, the efficiency reached a plateau, indicating the establishment of chemical equilibrium rather than a limitation by diffusion.

Considering both performance and practicality, the optimal condition for acetamiprid removal was

identified as a 3% sodium bicarbonate ($NaHCO_3$) solution applied for ten minutes. This treatment achieved approximately 75 ± 3% removal, which was significantly higher than that of the other cleaning agents ($p < 0.05$). Extending the washing time or further increasing the concentration did not provide additional improvement and could lead to tissue softening or nutrient loss.

In short, these results support a pseudo–first-order desorption model for pesticide removal. The reaction rate constant varies according to the chemical properties of the cleaning agent. Acidic conditions primarily promote desorption through protonation and limited hydrolysis. In contrast, alkaline and surfactant-based systems achieve faster kinetics because they combine chemical degradation, emulsification, and micelle encapsulation. The reduced benefit observed beyond 10 minutes or above 3% concentration indicates a shift from reaction-controlled to diffusion-controlled kinetics.

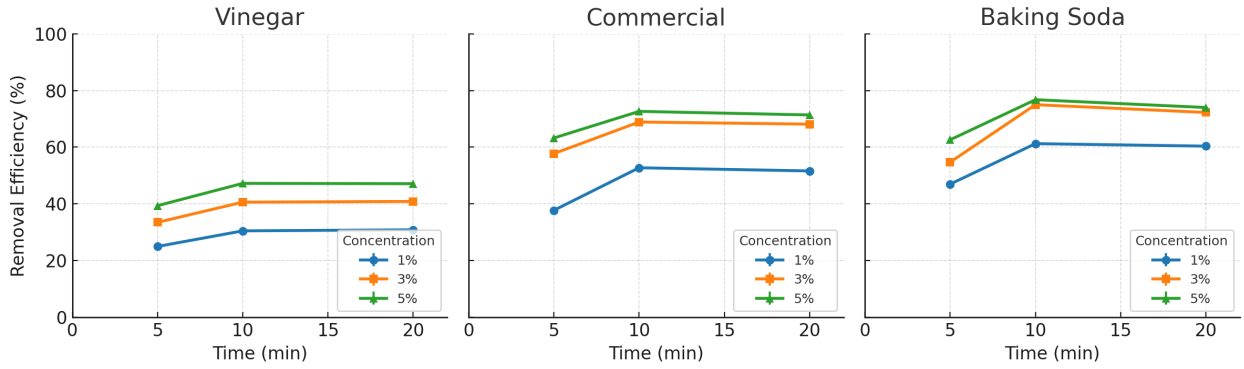


Figure 4 The evolution of removal efficiency on different cleaning time and concentration of cleaning agent vinegar, commercial and baking soda.

## 4. Conclusion

In summary, this study established a quantitative and reproducible approach for evaluating the removal efficiency of pesticide residues under controlled cleaning conditions. The method proved reliable and sensitive, allowing the assessment of multiple variables including cleaning agent, concentration, and exposure time and their combined effects on pesticide removal. Among the tested treatments, sodium bicarbonate (baking soda) exhibited the highest removal efficiency, followed by commercial detergent and vinegar, with simple water washing being the least effective. Statistical analyses (ANOVA, $p < 0.05$) confirmed that both concentration and cleaning time significantly influence removal efficiency, with a clear interaction between these two factors. The optimal cleaning condition identified in this study is a 3% $NaHCO_3$ solution with a 10 minute contact time, achieving up to 75% removal of acetamiprid residues. This provides an accessible and safe recommendation for household food preparation.

Future research should expand this methodology to encompass a broader range of pesticide classes with diverse chemical stability and polarity, as well as different vegetable and fruit matrices that vary in surface roughness and wax composition. In addition, integrating toxicological assessments will be essential to correlate residue reduction with actual health risk mitigation. In general, such efforts will strengthen the scientific basis for developing evidence-based food safety guidelines and enhance consumer confidence in managing pesticide residues during household cleaning.

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