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Review of Mechanical and Physicochemical Pretreatments in Essential Oil Extraction: Mass-Transfer Enhancement and Yield Optimization

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Abstract. Essential oils are volatile bioactive compounds widely used in pharmaceuticals, food preservation, cosmetics, and aromatherapy. Conventional hydrodistillation and steam distillation remain the primary recovery methods but suffer from long extraction times, moderate yields, and thermal degradation. Mechanical and physicochemical pretreatments address these limits by disrupting secretory structures, shortening diffusion paths, and enhancing mass transfer. From a chemical engineering perspective, this review synthesizes evidence published between 2010 and 2025 on particle-size reduction, ultrasound-assisted hydrodistillation, microwave-assisted hydrodistillation, steam explosion, instant controlled pressure drop, and cold pressing of citrus peels. Outcomes vary by matrix: in seeds such as celery, ultrasound-assisted hydrodistillation increases yield by nearly 50% compared with conventional hydrodistillation; in citrus peels, steam explosion accelerates extraction up to eightfold but reduces composition to limonene, while cold pressing preserves thermolabile aldehydes and esters crucial for fragrance. Instant controlled pressure drop applied to hyssop and Tagetes enhances yield, accelerates kinetics, and improves antioxidant indices through microstructural expansion confirmed by microscopy. In leaves and flowers including rosemary and lavandin, ultrasound- and microwave-based methods consistently shorten cycles while maintaining comparable chemical and sensory profiles. The addition of low-cost modifiers such as sodium chloride and optimized water-to-solid ratios further improves rosemary hydrodistillation without compromising oil quality. These findings highlight trade-offs among rate, yield, and composition. Standardized reporting of particle size, moisture content, and kinetic parameters is recommended to ensure reproducibility and cross-study comparison. Mechanical pretreatments thus provide a flexible framework to optimize essential oil extraction across industrial and bioengineering applications.

Introduction

Essential oils (EOs) are complex mixtures of volatile terpenoids and phenylpropanoids with wide-ranging applications in pharmaceuticals, food preservation, cosmetics, and perfumery [1]. Conventional extraction, principally hydrodistillation (HD) and steam distillation (SD), remains the industry workhorse, however, both are constrained by long cycle times and suboptimal recoveries attributable to mass-transfer barriers within the plant matrix [2]. The extraction efficiency in hydrodistillation is governed by two resistances in series: an internal resistance (R_int) arising from diffusion through the cuticle, cell walls, and secretory structures and an external film resistance

(R_film) associated with the interfacial boundary layer at the vapor-condensate interface. The contemporary mechanistic model explicitly considers intraleaf diffusion and convective film transfer and shows that both resistances shape the extraction curve and influence component selectivity during HD or SD [3]. The mass-transfer barriers governing essential oil release are illustrated in Figure 1.

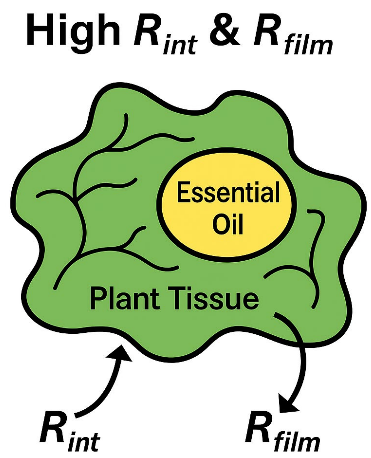


Fig. 1. Conceptual representation of mass-transfer barriers in essential oil extraction.

These barriers arise from cuticular layers, lignocellulosic cell walls, and the geometry of oil-bearing secretory tissues such as subdermal cavities, secretory canals, and glandular trichomes, which physically sequester EOs from the bulk fluid phase [4–7]. From a chemical engineering perspective, yield is governed by resistances in series: internal diffusion through disrupted tissues and external film transfer at the solid–liquid or solid–steam interface. Accordingly, pretreatments that rationally modify the microstructure and interfacial area should increase EO release by increasing the driving force-to-resistance ratio.

Mechanical approaches, such as particle size reduction milling, grinding, bruising or rolling, high-shear mixing, and mechanophysical intensification, such as ultrasound, steam explosion (SE), and instant controlled pressure drop (DIC), have been advanced to overcome these barriers [2,8–10]. Theoretically, comminution increases the accessible surface area and shortens the diffusion path; ultrasound generates cavitation microjets that perforate cells and secretory walls; and SE or DIC induce rapid decompression that puffs tissues, creating porosity that facilitates solvent or steam penetration [8–12]. However, higher mechanical or thermal energy inputs may also provoke volatilization losses during grinding, promote stable emulsions that complicate separation, and shift the relative abundances of monoterpenes, sesquiterpenes or phenylpropanoids, thereby altering aroma and bioactivity [1,2]. Thus, optimization must balance the dual objectives of maximizing yield and rate while preserving chemical integrity.

Empirical studies support both the promise and the limitations of these strategies. Ultrasound-assisted hydrodistillation (UAHD) often shortens the extraction time and increases the yield without major compositional drift when the amplitude and residence time are controlled [8,12,13]. Compared with untreated controls, thermomechanical pretreatments, particularly DIC, significantly increased the EO yield and antioxidant capacity in several matrices. Similarly, SE applied to citrus and other oil-bearing tissues improves mass transfer by rupturing resistant structures [9,11,12,14–16]. In contrast, the effect of particle size is strongly system dependent: while many studies report yield increases with controlled size reduction, others observe negligible or non-monotonic effects once external resistances dominate or volatilization offsets the gains [17–19]. These heterogeneous findings highlight the need for a framework that considers plant anatomy cavities, trichomes, moisture content, and process modality to explain and predict yield responses.

This review synthesizes evidence on mechanical approaches in EO extraction with two objectives: (1) to map mechanistic links between pretreatment parameters such as particle-size metrics, ultrasound power and duration, SE pressure and holding time, DIC temperature–time profiles, and plant-matrix attributes, including species, organ, moisture content, and extraction performance, such as yield and kinetics; and (2) to assess whether and how these approaches alter chemical composition, including marker analytes and broader volatile profiles. Four guiding research questions (RQs) are addressed:

RQ1. Which classes of mechanical pretreatment reproducibly increase the EO yield, and within what operating windows?

RQ2. How do plant-matrix attributes and extraction modalities such as HD, SD, hybrid, or thermomechanical processes influence yield and rate?

RQ3. To what extent do mechanical approaches shift the composition in terms of monoterpenes, sesquiterpenes, and phenylpropanoids, and are these shifts systematic or idiosyncratic?

RQ4. What reporting standards, including particle-size distributions, moisture basis, and kinetic descriptors, are required to enable reproducible optimization and cross-study comparability?

Guided by diffusion, film-transfer theory and secretory-anatomical evidence, this review formulates two working hypotheses (Hypothesis 1, H1, and Hypothesis 2, H2) to structure the analysis. H1 states that mechanical pretreatments produce a positive and practically significant increase over untreated controls up to an optimum intensity or size threshold, beyond which plateaus or reverts due to volatilization or matrix compaction. H2 states that, under controlled moisture and energy input, yield gains can be realized with limited and predictable compositional drift, thereby preserving functional quality. By explicitly linking microstructure to transport phenomena and composition, this review seeks to convert scattered reports into actionable guidance for laboratory and industrial practice and to motivate standardized protocols for future studies.

From a chemical engineering perspective, this review further emphasizes essential oil extraction as a separation process governed by mass transfer resistances and kinetic limitations. While existing reports often focus on yield or compositional outcomes, integrated evaluations that translate these findings into process engineering frameworks remain scarce. By positioning pretreatment strategies within the context of process intensification and energy-efficient separations, this work provides novel insights into how mechanical and physicochemical modifications can optimize yield, efficiency, and reproducibility across diverse plant matrices.

Methods

Study Design and Reporting

This article presents a narrative review that synthesizes empirical findings on mechanical approaches to essential oil (EO) extraction. This review integrates mechanistic insights with reported outcomes on yield, kinetics, and chemical composition. Literature searches were conducted in major scientific databases, including PubMed, Scopus, Web of Science, and Google Scholar, covering publications published between January 2010 and July 2025. Only peer-reviewed articles and full-text conference proceedings reporting experimental or comparative data were considered. Conceptual papers without experimental evidence, duplicate records, and studies outside the scope of EO extraction were excluded.

The narrative approach was selected to accommodate the methodological heterogeneity of bench-scale extraction studies, where process parameters, plant matrices, and evaluation criteria vary widely. This format enables a critical synthesis of available evidence and the identification of cross-cutting patterns rather than a meta-analysis of quantitative outcomes.

Eligibility criteria

This review included studies on plant matrices with secretory structures such as leaves, peels, seeds, aerial parts, and woods used for essential oil extraction. The mechanical pretreatments considered were particle size reduction by grinding, bruising, high-shear mixing, UAHD, SE, and DIC. Comparisons were made with untreated controls or alternative pretreatments at different intensities. The primary outcomes were essential oil yield and extraction kinetics, whereas the secondary outcomes included chemical compositions such as marker compounds and classes of monoterpenes, sesquiterpenes, and phenylpropanoids. Only experimental bench- or pilot-scale studies with quantitative results were included. The time window was from January 2010 to July 2025, which was limited to publications in English. Studies based on nonmechanical pretreatments, microwave-only processes, nonempirical papers, nonessential oil extracts, or unavailable full texts were excluded.

Information Sources and Search Strategy

Literature searches were carried out in Scopus, Web of Science Core Collection, PubMed, and Google Scholar through Publish or Perish, covering January 2010 to 31 July 2025. Additional records were identified by hand-searching reference lists of included studies and recent reviews. Boolean queries combine essential oil terms with extraction modalities, pretreatments, and outcomes. A representative Scopus query was "essential oil" OR "volatile oil" AND hydrodistillation OR steam distillation OR extraction AND particle size OR milling OR grinding OR comminution OR bruising OR high shear OR ultrasound OR sonication OR UAHD OR steam explosion OR instant controlled pressure drop OR DIC AND yield OR recovery OR extraction time OR kinetics OR composition. Search, screening, and documentation followed structured review practices, with syntax adapted to each database.

Data Extraction

Data were extracted independently by two reviewers via a piloted form. The information captured included plant matrix descriptors such as species, organs, anatomical notes, and geographical origins. The pretreatment parameters recorded were particle size, ultrasound power and duration, steam explosion settings, DIC cycles, high shear mixing speed, and bruising or rolling conditions. The extraction conditions included the method applied, liquid-to-solid ratio, heating or steam input, total time, and condenser characteristics when relevant. The material state was documented as the initial moisture content and any predrying treatment. The outcomes included essential oil yield in all reported units, extraction time, kinetic descriptors such as first-order rate constants when available, the composition of major constituents and compound classes, and quality indicators such as the refractive index. Design quality factors such as the number of replicates, control description, and statements on calibration or randomization were also extracted.

Results and Discussion

Characteristics of the Evidence Base

The search conducted between January 2010 and July 2025 identified comparative and optimization studies across five mechanical intensification strategies: UAHD, steam explosion, DIC, particle size reduction including cryogenic and ambient milling, and mechanical expression applied to citrus peels. Several studies have also modeled HD kinetics and process energetics. Collectively, these works span seeds such as celery and fennel; peels such as citrus and kumquat; leaves and flowers such as lavandin, rosemary, and thyme; and aerial herbs such as hyssop. This distribution enables a cross-matrix interpretation of how tissue type and gland morphology modulate the response to mechanical pretreatments [8,12,20–22]. Table 1 synthesizes how each approach primarily reduces either internal diffusion resistance (R_{int}) or external film resistance (R_{film}) and the expected consequences for the extraction rate, ultimate yield, and compositional selectivity.

Table 1. Mechanical approaches for essential oil extraction: targeted resistance and expected outcomes

Approach	Primary mechanical action	Effect on rate	Effect on ultimate yield	Impact on composition	Operating window/Reference
Conventional hydrodistillation (HD)	Heating + steam flow; no pretreatment	Baseline (slow)	Baseline (often incomplete)	Baseline profile	Use as reference condition
Ultrasound-assisted hydrodistillation (UAHD)	Cavitation microjets, microstreaming; boundary-layer renewal	↑ (faster; t90 ↓)	≈ (often unchanged)	≈ (usually preserved)	Typical: 20–40 kHz; watch overheating/foaming [10,23]
Instant controlled pressure drop (DIC)	Brief saturated steam exposure → instant ΔP to vacuum; swelling & micro-fracturing	↑	↑	≈/slight shift	Steam 0.1–0.7 MPa, 10–60 s; vacuum 5–10 kPa; 1–3 cycles [9,24]
Steam explosion (SE)	Rapid decompression through valve; shear-induced defibration and channel formation	↑↑ (often strongest)	Variable (↑ with risk of volatile loss)	May narrow toward dominant monoterpenes	Tune residence time to limit over-processing [25–27]
Particle-size reduction	Milling/grinding to expose glands and shorten diffusion distance	↑ at medium size; ↓ if fines	↑ at medium size; ↓ if fines	≈/drift if over-milled	Target ~0.5–2 mm; avoid fines to prevent channeling/compaction [28,29]

Note, R_int: internal resistance (intra-tissue diffusion barriers); R_film: external film resistance (interfacial boundary layer). The arrows (↑/↓/≈) indicate relative changes versus conventional hydrodistillation.

Effects of Mechanical and Physicochemical Pretreatments

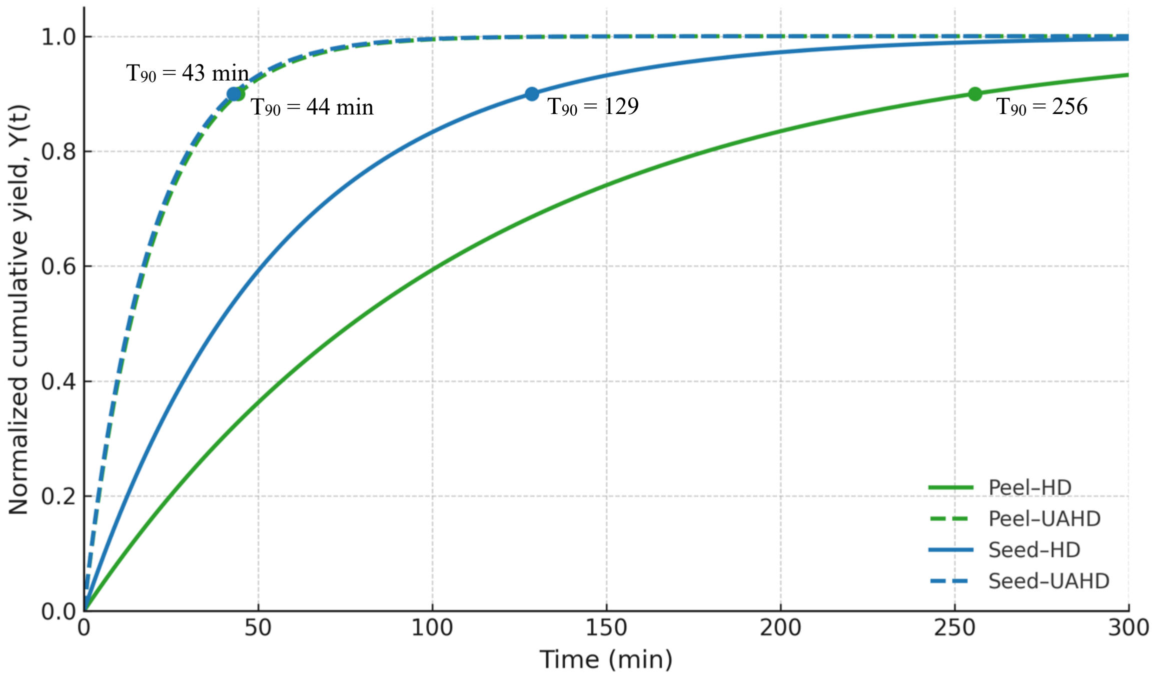
Ultrasound-assisted hydrodistillation (UAHD). Ultrasound generates cavitation that perforates secretory tissues, reducing internal diffusion resistance and accelerating extraction with minimal compositional drift. Across different plant matrices, UAHD consistently accelerated essential oil release and often increased yield without major changes in composition. A Taguchi design on celery seeds reported a 48.3% increase in yield under optimal sonication conditions of 50 min, 60% amplitude, and a pulsed regime. GC-MS and FT-IR analysis indicated high spectral similarity to conventional hydrodistillates, with near-infrared correlation values of 97.8%, confirming that rate enhancement was achieved with minimal selectivity change [8]. On thyme, an ultrasound pre-treatment followed by MW-HD cut extraction time by 72% and raised EO yield by ≈23%, while enriching thymol, versus HD [11].

In citrus kumquat peels, brief ultrasonic or microwave pretreatment prior to hydrodistillation significantly shortens the extraction time, with only modest variation in the relative abundance of oxygenated monoterpenes compared with that of hydrocarbons. This finding supports the broader observation that UAHD affects extraction kinetics rather than end-point compositional profiles. The quality index–based model proposed in that study further introduced an effective time-saving metric that may be applied for process benchmarking [30–32].

Comparative research on lavandin positioned ultrasound within the spectrum of emerging green intensification strategies. Compared with hydrodistillation and steam distillation, the ultrasonication and microwave methods reduced the extraction time from more than 200 minutes to approximately 30 minutes while maintaining comparable yields and sensorial attributes, again highlighting kinetics as the dominant advantage [21].

Mechanistically, cavitation generates localized microjets that perforate gland walls, reduce internal diffusion barriers, and renew the external boundary layer. Microstructural evidence of gland disruption has been documented in several intensified contexts; in thyme, ohmic-assisted thermal imaging revealed cell rupture patterns similar to those observed under sonication. These findings align with the characteristic acceleration toward the kinetic asymptote frequently observed in yield–time curves [33,34].

The kinetic parameters summarized in Figure 2 reinforce that UAHD markedly increases extraction rates relative to conventional HD, although the degree of improvement varies with matrix type.



Y(t): normalized cumulative yield (fraction of asymptotic yield, $Y_\infty = 1$)
t_{90}: time to achieve 90% of Y_∞

Fig. 2. Normalized extraction curves for HD and UAHD applied to *Citrus sinensis* peel and *Carum carvi* seeds.

For *Citrus sinensis* peel, previous research reported that UAHD increased the extraction rate constant nearly sixfold compared with HD, k = 0.052 vs 0.009 min^{-1}, reducing the time to 90% yield from approximately 256 minutes to 44 minutes [35]. In Carum carvi seeds, Kobus et al. (2014) reported that presonicated material reached comparable yields in 43 minutes compared with 129 minutes under HD, indicating a two-to-threefold reduction in time to target recovery [36]. Together, these results demonstrate that peel tissues with superficial oil glands benefit most strongly from cavitation-driven rupture, whereas seeds with thick integuments and high diffusion resistance show more moderate responses. In both cases, UAHD reduces internal and film mass-transfer resistance, leading to measurable time and energy savings in essential oil extraction.

Steam Explosion (SE) Pretreatment. SE ruptures lignocellulosic structures through rapid depressurization, dramatically increasing mass transfer but often narrowing the volatile profile. UAHD accelerates extraction through cavitation-induced microstreaming and gland wall disruption in liquid media, whereas SE intensifies at the solid matrix scale by subjecting plant tissues to high-pressure steam followed by instantaneous decompression. This abrupt transition results in autoevaporation, microfractures, and mechanical defibration, which rupture oil glands and shorten internal diffusion paths.

In orange (*Citrus sinensis*) peels, study reported optimized SE at 170 °C, 8 bar, and 240 s and reported that the same peel mass required nearly eight times longer to process under HD [12]. Gas chromatography–mass spectrometry revealed a marked enrichment of limonene, with SE extracts composed almost entirely of limonene compared with 77% in HD extracts and a corresponding loss of minor oxygenated volatiles. These findings illustrate the trade-offs inherent to SE: substantial time savings and simplified composition, favorable for biorefinery and solvent applications that prioritize limonene, but potentially suboptimal for fragrance and flavor industries where trace constituents are essential to aroma quality.

SE belongs to the broader family of thermomechanical treatments exemplified by DIC, which expand the matrix, rupture cell walls, and accelerate oil release within minutes rather than hours. Scanning electron microscopy and comparative process studies consistently attribute these effects to tissue expansion and reduced mass-transfer resistance. Recent reviews of DIC confirm these benefits and highlight its scalability, while also noting that elevated temperatures and pressures can bias composition toward hydrocarbon monoterpenes at the expense of oxygenates[11,37]. Taken together, the evidence supports the use of SE or DIC when throughput and limonene-rich fractions are prioritized, whereas gentler or staged methods such as HD, UAHD, MAHD, or fractionation remain preferable when compositional richness is the primary goal.

Instant Controlled Pressure Drop (DIC). DIC creates controlled microstructural expansion, improving yield and kinetics while maintaining more stable composition than SE. Unlike SE, which achieves rapid extraction primarily through severe thermomechanical disruption, often at the expense of compositional diversity, DIC provides more controlled pressure-based intensification. By coupling short-duration high-temperature steaming with instantaneous autovaporization, DIC retains most of the rate and yield advantages of SE while exerting a milder impact on the volatile profile. This makes DIC particularly valuable in contexts where both extraction efficiency and compositional integrity are critical.

DIC integrates brief exposure to high-pressure saturated steam followed by immediate decompression into vacuum. This sequence provokes autovaporization, rapid cooling, and explosive tissue expansion, leading to cell-wall rupture, increased porosity, and reductions in both internal and film mass-transfer resistance. Mechanistic overviews consistently describe these thermomechanical effects and their benefits for volatile release and subsequent hydrodistillation [11]. In Hyssopus officinalis, optimization via response-surface methodology demonstrated that DIC outperformed HD, ultrasound-assisted extraction, and Soxhlet extraction in a four-way comparison, resulting in higher apparent recovery, SEM-verified tissue expansion and rupture, and improves antioxidant indices relative to HD [22].

When DIC-HD was applied as a pretreatment immediately prior to HD (DIC-HD), the method produced quantified gains in spices. In cardamom seeds, DIC-HD increased the essential-oil yield from 2.52% under HD to 4.43%, with complementary improvements in antioxidant measures. The study provided a detailed description of the cycle, including vacuum, steam hold, and instant pressure drop, which corresponded with the microstructural unlocking evident in SEM images [9]. Taken together, the DIC literature indicates three consistent findings relevant to essential-oil extraction: robust improvements in rate and yield compared with baseline methods, moderate compositional shifts that preserve aroma profiles more faithfully than SE, and a validated mechanistic explanation centered on tissue expansion and cell wall rupture, which directly supports faster mass transfer [11,22].

Particle-Size Reduction and Mechanical Comminution. Comminution increases surface area and shortens diffusion paths, though excessive grinding may cause volatile loss or matrix compaction. While DIC illustrates how thermomechanical expansion can unlock oil glands and shorten diffusion paths at the tissue level, similar principles operate at the bulk scale through particle size reduction, where the degree of comminution directly controls both internal diffusion distances and bed permeability during HD.

The particle size simultaneously governs the diffusion path length and hydraulic behavior (Table 2). This reduction improves mass transfer up to an optimum value, but excessive fines increase compaction and channeling, impeding vapor–solid contact and reducing efficiency. Evidence from supercritical and subcritical packed-bed operations confirms that overly fine particles compromise drainage and flow distribution, a principle that translates directly to HD and SD of milled botanicals [38,39].

Clove buds (*Syzygium aromaticum*) exhibit this balance. In a controlled HD study, ground buds produced approximately double the oil yield of whole buds (14.3% and 7.14%), confirming that comminution shortens diffusion paths and accelerates release. However, excessive grinding disturbs steam circulation and slows extraction, highlighting the hydraulic penalty of excessive fines [18].

For fennel seeds (*Foeniculum vulgare*), a factorial design optimized HD and SD while testing CM as a pretreatment. Moderate CM for 3–5 min increased the yield by up to 18% and shortens the extraction time, whereas prolonged CM for 7 min reduced the yield, which was attributed to excessive rupture of the oil ducts, volatilization losses, and bed compaction. The same study proposed an optimal particle size of 315–500 µm for maximizing recovery [40].

Practical implications are clear: intermediate grinding that exposes glands without producing fines is generally optimal. Yield–size curves should be verified for each matrix, as overcomminution can alter the chemical balance and impair process hydraulics. When aroma preservation is critical, ultrafine or prolonged CM should be avoided unless validated in pilot trials. In contrast, moderate premilling is often beneficial for seeds and buds, provided that steam permeability is maintained [18,38,39].

Table 2. Effects of the particle size strategy on the essential oil yield and composition

Plant (part)	Milling strategy (screen size/cryo vs ambient)	Extraction (HD/SD/UA HD)	Change in yield vs whole (%)	Noted compositional effects	Ref.
Clove buds (*Syzygium aromaticum*)	Ambient grinding to ~0.6 mm vs whole buds ~2 cm	HD (Clevenger, 6 h)	+100% (14.3% vs 7.14%)	Eugenol decreased 87.39→68.73%; cyperene increased 7.22→20.52%; authors note overgrinding can disturb steam circulation and slow extraction	[18]
Fennel seeds (*Foeniculum vulgare*)	Cryomilling (CM) 5 min vs no CM baseline (HD 120 min, S/L = 1:10); also CM 3 min + HD 80 min	HD	+18% (6.49% vs 5.50%) at CM 5 min + HD 120 min; +9% (6.00% vs 5.50%) at CM 3 min + HD 80 min with 20 min time saving	GC–MS showed no qualitative differences among methods; CM samples had higher volatile amounts; prolonged CM (7 min) reduced yield; literature indicates optimal particle band 315–500 µm	[40]

As summarized in Table 2, the effect of particle size manipulation on EO recovery is highly matrix dependent and nonlinear. In *Syzygium aromaticum*, ambient grinding increased yield but altered the chemical balance, whereas in *Foeniculum vulgare*, moderate CM improves yield and kinetics, with excessive CM reducing efficiency. Collectively, these findings reinforce that intermediate milling rather than extreme fineness is most effective for balancing yield maximization with compositional integrity.

Hydrodistillation Modifiers: Salt Addition and Moisture Conditioning. Ionic-strength adjustment and moisture optimization alter matrix swelling and partitioning behavior, enabling faster hydrodistillation with preserved quality. In addition to structural and thermomechanical pretreatments, the efficiency of HD can also be shaped by medium composition and raw material conditioning, particularly salt addition and moisture control. These factors act through physicochemical pathways, complementing mechanical disruption by altering phase equilibria, swelling behavior, and steam permeation.

For rosemary (*Rosmarinus officinalis*), an orthogonal design demonstrated that adding 5% NaCl in combination with an optimized water-to-solid ratio significantly increased the oil yield, which was fitted well to a first-order kinetic model for Y(t). The same study reported that NaCl shortens the onset of oil rise and lowered the distillation temperature, confirming that phase behavior shifts rather than that tissue rupture alone drove these effects [41,42]. The moisture content further determines the extraction efficiency: overdried material is released less by collapsing oil ducts, whereas adequate hydration enhances swelling and permeability. These interactions require case-specific optimization, and rosemary factorial designs highlight the value of preliminary screening strategies such as the Plackett–Burman or Taguchi methods before scale-up [41].

The parametric responses illustrated in Figure 3 emphasize that rosemary oil recovery strongly depends on both ionic and hydration effects. Moderate NaCl addition increased yield and reduced onset time, whereas excessive NaCl addition suppressed recovery, which was in line with salting-out behavior. A water-to-solid ratio of 1:3 produced the highest yield (1.16%) compared with 1:1 or 1:2, with extraction curves showing an excellent fit to first-order kinetics ($R^2 > 0.95$) and an asymptotic yield near 2.67% [41]. Comparable trends were observed in the ohmic-assisted HD of *Vitex pseudonegundo*, where NaCl acted as an electrolyte to accelerate heating, increasing the rate constant more than fivefold compared with that of conventional HD.[43–45]. Cross-technology evidence reinforces these findings. Ultrasound pretreatment of Citrus sinensis peel reduced the time to reach 90% yield from over 250 minutes under HD to approximately 44 minutes under UAHD while maintaining compositional fidelity [46]. Steam explosion of orange peels accelerated eightfold but shifted the composition toward limonene dominance and reduced the contents of minor constituents.

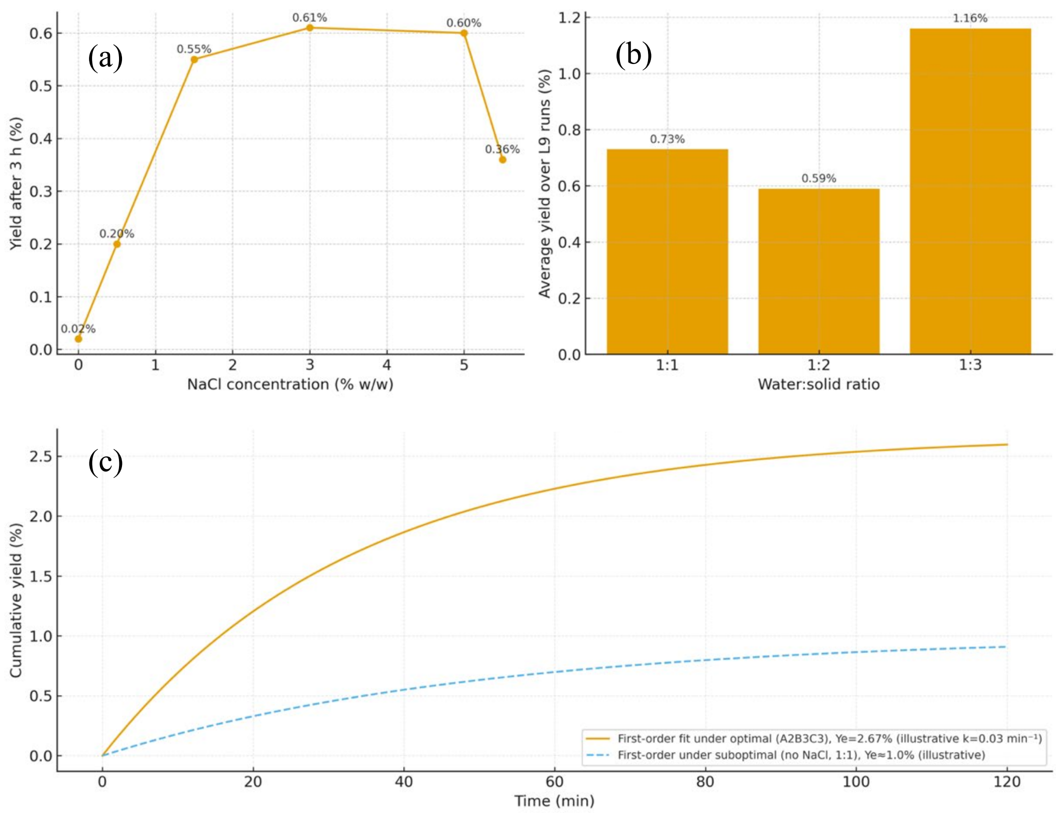


Fig. 3. Parametric sensitivity of rosemary hydrodistillation: (a) NaCl vs oil yield (3 h HD), (b) Water: solid ratio vs average yield, (c) First-order kinetic model form [41]

As summarized in Table 3, modifiers and pretreatments consistently reduce the mass-transfer resistance, although their outcomes vary in magnitude and selectivity. In rosemary, NaCl and water:solid optimization improves yield with predictable kinetics [41]. Ohmic-assisted HD of Vitex pseudonegundo resulted in a more than fivefold increase in the rate constant [42].

Table 3. Comparative effects of modifiers and pretreatments on essential oil extraction

Plant (part)	Modifier/Pretreatment	Extraction method	Key effect on yield/time	Compositional notes	Ref.
Rosemary (*Rosmarinus officinalis*)	NaCl (0–5.5% w/w); water:solid ratio (1:1–1:3)	HD (Clevenger)	Yield ↑ to 1.16% at 1:3 ratio; yield optimized near 5% NaCl; followed FK kinetics (R^2 > 0.95, Ye 2.67%)	No qualitative shifts, only phase behavior effect	[41]
Vitex pseudonegundo	NaCl electrolyte in ohmic heating	Ohmic-assisted HD	Extraction rate constant 5.05× that of conventional HD; reduction in extraction temperature and time	Electrolyte-enhanced heating; no quality degradation noted	[42]
Citrus waste (peel/pulp)	Ultrasonic pretreatment	Ultrasound-assisted HD	Yield ↑ by 33% and antioxidant activity improves (44%)	No major component loss; antioxidant compounds extracted	[46]
Orange peel (*Citrus sinensis*)	Steam Explosion (170 °C, 8 bar, 240 s)	SE-HD	Extraction 8× faster than HD; limonene increased to 100% vs 77% in HD; fewer minor compounds	Composition narrowed to limonene-rich fraction	[12]

Ultrasound pretreatment of citrus residues improves yield and antioxidant recovery without compositional loss [46], whereas steam explosion of orange peels accelerated extraction eightfold but biased the profile toward limonene [12]. Collectively, these findings demonstrate that salt addition, moisture management, and physical intensification act through complementary mechanisms to improve essential oil recovery, and their application should be guided by whether the priority is rate, yield, or compositional fidelity.

Mechanical Expression of Citrus Peels by Cold Pressing. Mechanical rupture of oil sacs releases volatiles without heat, preserving thermolabile aldehydes and esters essential for fragrance-quality oils. Unlike thermally driven HD or pressure-assisted pretreatments, cold pressing is a purely mechanical technique widely adopted in the flavor and fragrance industry for citrus peel oils. This method ruptures flavedo oil glands under pressure and releases volatiles with minimal heat input, thereby preserving aldehydes, esters, and terpenoids that are often degraded or lost during distillation.

A comparative study on *Citrus paradisi* (grapefruit) and *Citrus grandis* (pummelo) demonstrated that cold-pressed oils provided higher yields than HD did. Compared with 96.1% distilled oil, cold-pressed grapefruit oil contained 92.8% limonene, indicating a slight shift toward lighter hydrocarbons while retaining more oxygenated compounds that contribute to antimicrobial and antioxidant activity [47].

Another investigation across several *Citrus* species, including Valencia orange, Ponkan, and Eureka lemon, combined GC–MS with direct comparisons of HD and cold pressing. The results revealed that cold-pressed oils better preserved selected terpenes and oxygenated constituents, particularly those of lemon varieties, highlighting method-dependent differences in aroma quality. In these cases, compositional preservation rather than large differences in yield was the main advantage [48].

Cross-Cutting Themes

Kinetics Modeling and Rate Selectivity Trade-offs. Extraction kinetics provide a unifying framework for assessing how mechanical and physicochemical intensification strategies influence both the rate of oil release and the selectivity of compositional profiles. First-order models consistently describe HD, yet the time required to reach 90% of the asymptotic yield (t90) varies substantially among pretreatments [41]. The trade-off between speed and chemical fidelity is therefore central to determining whether a process is better suited for industrial biofuels, which prioritize terpene recovery, or for flavor and fragrance applications, which depend on minor oxygenated volatiles.

Case comparisons highlight this balance. Compared with HD, a dual-function UAHD prototype reduced the isolation time of Citrus sinensis peel by 83% while maintaining a terpene-rich profile and minimizing thermal artifacts. The study also reported linearized first- and second-order model fits that enabled rate comparisons between modes [35]. In contrast, SE delivers even greater time gains, with HD requiring eight times longer for the same peel mass, but GC–MS revealed a strong narrowing of composition, with limonene at 100% under SE compared with 77% under HD and a substantial loss of minor volatiles [12]. DIC represented an intermediate pattern: response surface optimization of

Hyssopus officinalis resulted in higher yields than HD, ultrasonication, and Soxhlet, as supported by SEM evidence of tissue expansion, reduced diffusion resistance, and improves phenolic indices. The volatile shifts were moderate, in contrast with the more pronounced narrowing observed under SE [22].

Overall, these findings indicate that pretreatments can be positioned along a rate–selectivity continuum. UAHD typically involves rapid extraction with broad compositional preservation. DIC offers comparable acceleration with modest compositional adjustments driven by microstructural unlocking. SE achieves the fastest extraction but with significant narrowing of volatile profiles. First-order modeling provides a practical basis for comparing rate constants and t90 across these regimes, guiding the selection of conditions that align with product objectives. Fragrance applications benefit from preserving compositional richness, whereas biofuel or biorefinery processes may accept narrower profiles in exchange for higher throughput [12,22,35,41].

Integrative Framework for Selecting Mechanical Pretreatments. Integrating comparative evidence across seeds, peels, and leaf–flower matrices shows that the optimal pretreatment is highly matrix dependent, with each strategy offering distinct advantages and trade-offs in kinetics, yield, and compositional fidelity. The matrix-specific outcomes are summarized in Table 4.

Seeds, as hard matrices with small embedded glands, benefit most from UAHD since acoustic cavitation shortens diffusion paths and accelerates gland rupture. In celery seeds, UAHD increased yield by approximately 48% compared with HD while reducing energy use, demonstrating a clear rate–yield advantage for dense matrices. Intermediate grinding is recommended to expose the oil ducts, but overpulverization should be avoided because it compacts the bed and promotes premature volatilization. Compared with HD alone, CM may be tested at short durations, as fennel seed studies have shown that brief CM improves yields, whereas longer exposure reduced the benefit. When equipment is available, DIC can further unlock the cellular microstructure, delivering higher yields than HD, UAHD, and Soxhlet in Hyssopus officinalis. SEM confirmed that tissue expansion and antioxidant indices improves, which is particularly useful when the coextraction of phenolics is desirable [8,40].

Table 4. Reported effects of mechanical and physicochemical pretreatments on essential oil yield, kinetics, and composition across different botanical matrices.

Plant (part)	Mechanical approach	Extraction	Key setting	Outcome	Composition	Ref.
Apium graveolens (seeds)	UAHD (pretreat)	HD	50 min; 60% amplitude; pulsed	+48.3% yield vs HD; faster	High spectral similarity to HD	[8]
Citrus orange (peel)	Steam explosion	—	170 °C; 8 bar; 240 s	8× faster than HD	Fewer GC–MS peaks; limonene ↑ (100%)	[12]
Hyssopus officinalis (herb)	DIC (stand-alone)	—	1 bar; 100 s; 12 cycles (optimum)	Higher yield vs HD/UAE/Soxhlet	Antioxidant ↑; SEM shows rupture	[22]
Tagetes lucida (peels/leaves)	DIC pretreat → HD	HD	Optimized DIC-HD	4.43% vs 2.52% (HD)	Acceptable profile for flavor	[9]
Lavandin (*Lavandula × intermedia*)	Ultrasound/microwave	SD/HD	—	Time ↓ dramatically; yield ~similar	Sensorial profiles comparable	[21]
Foeniculum vulgare (seeds)	Ambient vs cryogenic milling	HD	—	Ambient-milled > cryomilled yield	Cryomilling may prevolatilize	[40]
Clove buds	Grinding + wetting	SD	—	Yield ↑ vs whole buds	—	[12]
Rosemary	NaCl modifier	HD	5% NaCl; W:S≈1:3	Yield ↑; faster onset; first-order fit	No adverse quality noted	[41,42]
Citrus (peels)	Cold press vs HD/MAHD vs SFME	—	—	Avg yields: 2.85% (CP); 2.87% (HD/MAHD); 5.29% (SFME)	CP preserves cold profile; SFME fastest	[49]
Citrus peels	Solar-thermal HD (PTC)	SD	Field pilot	Yields comparable to gas-fired; system η 55%	Limonene 90%	[50]

Peels from citrus flavedo with large vesicles near the surface are best processed by cold pressing when fragrance-grade oils are the target, as this method preserves thermolabile top notes. In orange and tangor, cold-pressed oils maintained a fresh citrus aroma and prevented the off-flavor formation observed under HD. When higher throughput is required without compromising sensory quality, UAHD on orange peels accelerates extraction and increases yield compared with HD. For maximum speed or when a limonene-rich profile is the objective, SE at 170 °C, 8 bar, and 240 min achieved extraction nearly eight times faster than HD and shifted the composition toward limonene with the loss of minor constituents, representing a deliberate selectivity trade-off. Solar thermal steam distillation with parabolic-trough collectors offers an additional route to decarbonize HD while maintaining yields and limonene content comparable to those of gas-fired systems, making it suitable for peel-processing operations in regions with strong solar resources [10,51].

Leaves and flowers such as rosemary, thyme, and lavandin, which are rich in glandular trichomes, respond well to MAHD and UAHD–MAHD hybrids. These methods typically reduce cycle time while preserving yields and compositions comparable to those of HD. In rosemary, MAHD achieved the same yield in 75 minutes compared with 210 minutes for HD. A comparative study reported similar chemical profiles between HD and microwave hydrodiffusion and gravity but with much shorter isolation times in the microwave process. Reviews of UAHD confirm that significant time savings are achievable without compromising quality when the acoustic intensity and temperature are controlled. Regardless of the heating method, small additions of NaCl (approximately five percent) and optimized water-to-solid ratios improve HD kinetics and follow first-order models, providing a simple framework for predicting the time to target yield. Milling practices are equally important: excessive fines increase bed compaction and resistance, whereas moderate comminution, which exposes trichomes without producing dust, performs best [10,51,52].

In practice, seeds respond best to UAHD combined with intermediate grinding, with CM occurring only at short durations when UAHD reaches a performance plateau. DIC is an additional option where equipment is available and when rapid throughput or antioxidant retention is desired. For citrus peels, cold pressing remains the preferred choice for perfumery and flavor products because of its ability to preserve delicate aroma compounds. UAHD is appropriate for accelerating kinetics while preserving composition, DIC is effective for high throughput with moderate shifts, and SE is suitable when the limonene concentration and minimal residence time are prioritized. For rosemary, thyme, and lavandin, MAHD or UAHD reduce time and energy use while maintaining sensory quality. In contexts where advanced hardware is unavailable, HD can still be optimized by adjusting the NaCl concentration and water-to-solid ratio. Across all the matrices, fitting first-order models to pilot data provides estimates of the kinetic constant (k) and asymptotic yield ($Y\infty$), enabling the optimization of operating conditions according to product goals, whether maximizing speed, preserving minor volatiles, or lowering the carbon footprint of processing [8,41,50].

Conclusion

Mechanical and physicochemical pretreatments enhance mass transfer and yield in essential oil extraction, although their performance remains highly dependent on the plant matrix and must be aligned with specific product objectives. Ultrasound and microwave methods consistently accelerate extraction and preserve chemical fidelity. Instant controlled pressure drop increases yield and antioxidant capacity with only moderate compositional shifts. Steam explosion achieves the fastest release but reduces volatile diversity. Cold pressing maintains thermolabile components that are crucial for citrus-derived products. Overall, these findings demonstrate that pretreatments function as controllable levers that modify internal and external resistances and enable targeted process intensification. Future research should prioritize standardized kinetic descriptors, harmonized reporting of particle size and moisture content, and the integration of energy and environmental metrics to support the transition from laboratory advances to scalable and sustainable industrial applications.

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Conflict of interest

The authors declare that there are no conflicts of interest regarding the publication of this review article. The work was conducted independently, and no financial or personal relationships influenced the preparation, analysis, or interpretation of the manuscript.

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