The Impact of Drying Methods on the Phytochemical Profile of Citrus sinensis Essential Oils: A Review

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Abstract- The effect of post-harvest drying techniques on the phytochemical composition of Citrus sinensis essential oils has been extensively studied. This review synthesizes findings from a thesis investigating how different drying durations influence the yield and chemical constituents of the essential oil extracted from Citrus sinensis leaves. The study reveals that drying leads to both qualitative and quantitative changes in the oil's monoterpenoid and sesquiterpenoid content. Major components such as α-terpinolene, β-elemene, limonene, citronellal, and citral exhibit notable variations based on the drying period. The findings suggest that drying methods play a crucial role in optimizing essential oil yield and composition, with implications for industrial and therapeutic applications.

Indexed Terms- Post-harvest drying, Phytochemical composition, Citrus sinensis, Essential oils, Drying techniques, Monoterpenoids, Sesquiterpenoids, α-Terpinolene, β-Elemene, Limonene, Citronellal, Citral, Chemical constituents

I. INTRODUCTION

Essential oils are intricate mixtures of volatile secondary metabolites that occur naturally in various plant species. These compounds play pivotal ecological roles, including plant defense mechanisms against herbivores and pathogens (Shawe, 1996; Lawrence, 2000), allelopathic interactions with neighboring plants (Lawrence, 2000), and attracting pollinators to ensure reproductive success. Among the plant families known for their rich essential oil content is the Rutaceae family, which includes *Citrus sinensis*, commonly referred to as sweet orange (Usman et al., 2016).

The diverse bioactive compounds present in essential oils have made them indispensable in multiple industries In the food industry, they serve as natural flavor enhancers and preservatives (Gupta et al., 2010; Martín et al., 2010). In pharmaceuticals, their antimicrobial, antioxidant, and anti-inflammatory properties make them valuable components in drug formulations (Campelo et al., 2011; Kumar et al., 2011). Additionally, the cosmetic industry leverages their aromatic qualities and skin-nourishing benefits for a wide range of products (Frassinetti et al., 2011).
Despite their widespread applications, the composition of essential oils can vary significantly due to several factors. Plant species, geographical origin, harvest time, and post-harvest processing methods all influence the phytochemical profile of these oils. Of particular interest is the role of drying methods, which can profoundly affect the quality and efficacy of essential oils by altering the volatilization and degradation of bioactive compounds. Commmon drying techniques include air-drying, sun-drying, and warm-air drying, each with unique effects on the chemical composition of the resulting essential oil.

This review focuses specifically on how different drying techniques impact the monoterpenoid and sesquiterpenoid composition of *Citrus sinensis*essential oils. By analyzing studies that examine the relationship between drying durations and changes in oil yield and chemical transformations, this work aims to provide actionable insights into optimizing post-harvest processes. Such optimization ensures the preservation of both the quality and therapeutic potential of *Citrus sinensis*essential oils.

II. ESSENTIAL OIL COMPOSITION AND YIELD

2.1 Impact of Drying on Oil Yield

One critical factor influencing the yield of essential oils from *Citrus sinensis*leaves is the method and duration of drying. Research has demonstrated that fresh leaves typically exhibit lower oil yields compared to dried leaves. For instance, oil yields from fresh *Citrus sinensis*leaves generally range between 0.09% and 0.22%. Conversely, dried leaves show

significantly higher yields, with some studies reporting up to 0.42% (Usman LA, et al, 2016) .
A detailed study revealed that the highest oil yield (0.37%) was achieved when leaves were dried for five days during the dry season. This finding underscores the importance of environmental conditions, such as humidity and temperature, in determining optimal drying periods. On the other hand, the lowest yield (0.09%) was observed in fresh, undried leaves, highlighting the necessity of some form of moisture reduction to enhance oil concentration.

The variations in oil yield can largely be explained by the process of moisture loss during drying. As water evaporates, the concentration of essential oils within the plant material increases, leading to higher extraction efficiency. However, it is important to note that excessive drying may not always result in increased yields. Prolonged exposure to heat or extended drying times can cause the volatilization of certain essential oil components, thereby reducing the overall yield. For example, drying for only one day resulted in relatively low oil yields (0.13–0.16%), while drying for two to three days produced a more pronounced increase in yield.

These findings suggest an optimal window for drying duration, where the balance between moisture loss and compound preservation is maintained. Identifying this window is crucial for maximizing the yield and quality of *Citrus sinensis*essential oils. Furthermore, understanding the specific impacts of different drying methods on individual bioactive compounds will help refine post-harvest practices tailored to preserve the unique characteristics of these oils.

2.2 Monoterpenoids and Sesquiterpenoids: Key Components of *Citrus sinensis*Essential Oils
Monoterpenoids and sesquiterpenoids constitute major classes of bioactive compounds found in *Citrus sinensis*essential oils. Monoterpenoids, such as limonene and linalool, are characterized by their high volatility and contribute significantly to the aroma and biological activity of the oil. Sesquiterpenoids, like β-caryophyllene and α-humulene, tend to be less volatile but possess potent anti-inflammatory and antimicrobial properties.

Drying methods and durations can alter the relative proportions of these compounds in the essential oil. For example, shorter drying periods might favor the retention of highly volatile monoterpenoids, whereas longer drying times could lead to their partial loss through evaporation. Meanwhile, sesquiterpenoids, being less volatile, may remain stable even under prolonged drying conditions. However, excessive heat during drying could still degrade these compounds, affecting the overall quality of the oil.

To illustrate, a study comparing air-drying and warm-air drying found that air-dried samples retained higher levels of limonene compared to warm-air dried samples. This difference was attributed to the gentler nature of air-drying, which minimized thermal degradation of sensitive compounds. Similarly, another investigation highlighted the importance of controlling drying temperatures to prevent the breakdown of sesquiterpenoids like β-caryophyllene, which are critical for the therapeutic value of the oil.

2.3 Practical Implications for Post-Harvest Processing
The results discussed above carry significant implications for the development of standardized post-harvest protocols for *Citrus sinensis*essential oils. Manufacturers and producers must carefully consider the type of drying method employed, along with its duration and associated environmental parameters, to achieve the desired balance between oil yield and compositional integrity.

For instance, air-drying at ambient temperatures may be preferable for preserving volatile monoterpenoids, while controlled warm-air drying at moderate temperatures could be used to stabilize sesquiterpenoids without excessive volatilization. Additionally, seasonal variations should be accounted for, as differences in climatic conditions can influence the effectiveness of drying techniques.

In conclusion, understanding the interplay between drying methods and the phytochemical profile of *Citrus sinensis*essential oils is essential for ensuring consistent product quality. Future research should focus on identifying novel drying technologies that minimize losses of bioactive compounds while enhancing oil yield. Such advancements will not only improve the economic viability of essential oil production but also expand their applications across various industries.

2.2 Chemical Changes in Essential Oils Due to Drying
Drying significantly influenced the qualitative and quantitative composition of monoterpenoids and sesquiterpenoids in *Citrus sinensis* essential oils. The study identified a total of 64 monoterpenoids, constituting between 54.9% and 90.2% of the oils, while sesquiterpenoids accounted for 6.9% to 27.5%. The drying process led to both the loss of some volatile compounds and the formation of new ones due to enzymatic or oxidative transformations.

Key findings include:

- Monoterpenoids: Limonene, a major constituent of *Citrus sinensis* essential oil, showed a decrease in concentration as drying progressed. However, α-terpinolene levels increased in leaves dried for three to four days.
- Sesquiterpenoids: β-Elemene was the most abundant sesquiterpenoid, though its concentration fluctuated depending on drying time. Notably, sesquiterpenoids generally increased in concentration as drying prolonged, except for the five-day dried leaves, which exhibited a reduction due to volatilization.

Several new compounds were identified in the dried leaves but were absent in fresh samples. This suggests that some terpenoids might exist in glycosidic forms in fresh leaves and are released upon drying due to the breakdown of glycosidicbonds.

The effects of drying on individual compounds are summarized in the following observations:

- Fresh leaves contained high levels of α-fenchene (12.7%), which was significantly reduced in dried leaves.
- Citronellol and citral, known for their antimicrobial properties, were more concentrated in leaves dried for two to three days.
- β-Caryophyllene, a sesquiterpenoid with insecticidal properties, showed an increasing trend in oils obtained from dried leaves.

These changes indicate that the drying process not only affects the yield but also modifies the chemical composition, potentially enhancing or reducing the oil's therapeutic properties.

The study highlights the significant impact of drying on the phytochemical profile of *Citrus sinensis* essential oils. While drying generally enhances oil yield by reducing moisture content, it also alters the chemical composition, with some compounds increasing and others decreasing due to volatilization or degradation. The findings emphasize the need for optimizing drying techniques to balance oil yield and chemical stability for industrial applications in pharmaceuticals, cosmetics, and food processing.

Future research should explore the effects of different drying temperatures and humidity levels to refine post-harvest practices for preserving the quality of *Citrus sinensis* essential oils. Additionally, advanced analytical techniques such as FTIR and NMR could be used to gain deeper insights into the molecular changes induced by drying.

III. EFFECT OF DRYING ON MAJOR PHYTOCHEMICALS

The phytochemical profile of *Citrus sinensis*essential oils undergoes significant changes depending on the duration and method of drying. These variations are primarily driven by two key processes: the evaporation of highly volatile components and the potential formation of new compounds during drying. Understanding these dynamics is crucial for preserving the quality and efficacy of the essential oil.

3.1 Limonene Variation Due to Seasonal Differences
Limonene, a dominant monoterpenoid in *Citrus sinensis*essential oils, demonstrates marked variability influenced by seasonal factors. During the rainy season, limonene concentrations range from 8.2% to 11.0%, while they significantly drop to 0–7.0% during the dry season. This decline is particularly pronounced in fresh leaves harvested in the afternoon during the dry season, where limonene levels may be entirely absent.

The seasonal fluctuations in limonene levels can be attributed to environmental conditions such as temperature and humidity, which play critical roles in the biosynthesis and stability of monoterpenoids.

Higher temperatures and lower humidity during the dry season likely accelerate the volatilization of limonene, reducing its concentration in the plant material. These findings underscore the importance of considering harvest timing and climatic conditions when optimizing post-harvest processing methods.

3.2 Fluctuations in Citronellal and Citral Levels

Citronellal and citral, oxygenated monoterpenoids with notable biological activities, exhibit dynamic changes based on drying duration. Citronellal was found to be more abundant in leaves dried for two to three days, after which its concentration decreases due to oxidation and volatilization. Similarly, citral levels were higher in dried leaves compared to fresh samples, suggesting that drying may facilitate the conversion of precursor compounds into citral.

This phenomenon highlights the dual nature of drying—while it can lead to the loss of certain volatile compounds, it may also promote the formation of others through enzymatic or chemical transformations. The balance between these processes depends on the specific drying conditions employed, emphasizing the need for precise control over temperature, humidity, and drying time.

3.3 Sesquiterpenoid Stability and Changes

Sesquiterpenoids, including β-caryophyllene and humulene, tend to remain relatively stable across different drying conditions, making them less prone to volatilization compared to monoterpenoids. However, α-sinensal, an oxygenated sesquiterpenoid, shows increased abundance in dried leaves compared to fresh ones. This suggests that oxidation processes occurring during drying enhance the formation of α-sinensal, likely through the transformation of its precursors.

Key observations regarding sesquiterpenoids include:

- β-Elemene : Its concentration fluctuates depending on drying duration, with peak levels observed in leaves dried for three days.
- β-Caryophyllene : Known for its insecticidal and antimicrobial properties, this compound exhibits stable levels regardless of drying conditions, indicating its resilience to degradation.
- Humulene : Levels of humulene remain relatively unchanged, further supporting its resistance to volatilization under typical drying scenarios.

These findings demonstrate that sesquiterpenoids, while generally stable, can still undergo subtle changes in response to drying, underscoring the complexity of their behavior during post-harvest processing.

IV. MECHANISMS BEHIND CHEMICAL CHANGES

The variations in the composition of *Citrus sinensis*essential oils can be explained by several underlying mechanisms, including volatilization, post-harvest metabolic activity, and oxidation/degradation. Each mechanism contributes uniquely to the final phytochemical profile of the oil.

4.1 Volatilization of Highly Volatile Compounds

Compounds with low molecular weights, such as limonene and α-pinene, are highly susceptible to volatilization during drying. This explains why their concentrations decrease significantly in dried leaves compared to fresh ones. Additionally, some sesquiterpenoids, like β-elemene, show minor reductions in concentration due to prolonged exposure to drying conditions. The extent of volatilization depends on factors such as temperature, airflow, and relative humidity, all of which should be carefully controlled to minimize losses of valuable volatile compounds.

4.2 Post-harvest Metabolic Activity

Certain compounds detected exclusively in dried leaves but not in fresh ones suggest the presence of post-harvest metabolic activity. For example, α-sinensal and citral are more prevalent in dried samples, likely due to the enzymatic hydrolysis of glycosidic precursors present in fresh leaves. This process releases bioactive compounds during drying, contributing to the enrichment of the essential oil's phytochemical profile. Optimizing drying conditions to maximize enzymatic activity could therefore enhance the yield of desirable compounds.

4.3 Oxidation and Degradation

Exposure to air and heat during drying induces oxidative changes in the essential oil composition. Oxygenated monoterpenoids, such as linalool and α-terpineol, exhibit reduced concentrations in dried leaves due to oxidative degradation. Conversely, some sesquiterpenoids, like β-caryophyllene oxide, are formed as oxidation products of β-caryophyllene, indicating ongoing chemical transformations during drying. While oxidation can lead to the loss of certain compounds, it can also generate novel derivatives with distinct biological properties.

These mechanisms collectively shape the phytochemical profile of *Citrus sinensis*essential oils. To maintain their integrity, drying protocols must strike a balance between minimizing undesirable losses (e.g., volatilization and degradation) and promoting beneficial transformations (e.g., enzymatic release and oxidation). Future research should focus on identifying optimal drying parameters that preserve the full spectrum of bioactive compounds while enhancing the overall quality of the essential oil.

In summary, the impact of drying on the phytochemical profile of *Citrus sinensis*essential oils is multifaceted, involving complex interactions between volatilization, enzymatic activity, and oxidation. By understanding these mechanisms, producers can develop tailored drying strategies to optimize the yield and composition of essential oils, ensuring their continued utility in food, pharmaceutical, and cosmetic applications.

V. IMPLICATIONS FOR INDUSTRIAL APPLICATIONS

Understanding the effects of drying on *Citrus sinensis* essential oils has significant industrial implications, as the changes in oil yield and composition influence its suitability for different applications.

Aromatherapy and Cosmetics

Essential oils are widely used in aromatherapy and cosmetic formulations due to their pleasant fragrances and potential therapeutic benefits. The study showed that drying methods can enhance or reduce the presence of key fragrance compounds such as limonene, α-terpinolene, and citral, which contribute to the citrusy aroma of *Citrus sinensis*oil. The optimization of drying techniques is crucial to ensuring the retention of these volatile aromatic compounds while minimizing losses due to oxidation and volatilization.

Pharmaceutical Applications

Essential oils from *Citrus sinensis* have demonstrated antimicrobial and insecticidal properties, primarily due to the presence of citronellal, citral, and β-caryophyllene. The study found that drying for two to three days resulted in higher concentrations of these bioactive compounds, enhancing the potential pharmaceutical applications of the oil. These findings suggest that controlling drying conditions could optimize essential oils for use in natural antimicrobial and insecticidal formulations.

Food Industry

Essential oils are commonly used as natural flavoring agents in food and beverages. The stability of key flavor compounds such as limonene and citral is crucial in maintaining the desired sensory characteristics of citrus-based products. The study showed that limonene content decreased with prolonged drying, highlighting the need to optimize post-harvest drying conditions to preserve the oil's flavorintegrity.

VI. CONCLUSION AND RECOMMENDATIONS

This review highlights that drying has a profound impact on the phytochemical profile of *Citrus sinensis* essential oils. The process increases oil yield by reducing moisture content but also alters the composition of monoterpenoids and sesquiterpenoids. Notably, drying results in reductions in limonene while enhancing concentrations of sesquiterpenoids such as β-elemene and α-sinensal.

Recommendations

1. Optimized Drying Conditions: Further research should explore optimized drying techniques that balance oil yield and composition to maximize the retention of key bioactive compounds.
2. Advanced Analytical Techniques: The use of Fourier Transform Infrared Spectroscopy (FTIR) and Nuclear Magnetic Resonance (NMR) should be employed to validate and quantify the chemical changes induced by drying.

3. Temperature and Humidity Effects: Investigations into how varying drying temperatures and humidity levels affect essential oil composition could help refine post-harvest practices for improved industrial applications.

By refining post-harvest drying techniques, industries can enhance the quality, stability, and efficacy of *Citrus sinensis* essential oils for applications in cosmetics, pharmaceuticals, and the food industry

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