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Research Article

Optimization, Chemical Profiling, and Bioactivity of Essential Oils from Citrus Peels Subjected to Different Drying Processes

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Abstract

Citrus processing generates large quantities of peel by-products that represent an abundant and renewable source of essential oils and other bioactive compounds. However, drying, a necessary pretreatment for storage and extraction, can significantly affect essential oil yield, chemical characteristics, and biological properties. This study aimed to investigate the effects of different drying methods and operating conditions on the extraction yield, physicochemical properties, and biological activities of essential oils obtained from peels of three Tunisian citrus species: *Citrus sinensis* (E1), *Citrus limon* (E2), and *Citrus aurantium* (E3). Physicochemical and thermophysical properties of the peels, including moisture content, porosity, and volumetric shrinkage, were first determined to evaluate their influence on drying behavior and oil extractability. Drying kinetics were then investigated using infrared, forced-convection, and open-air drying methods. A full factorial experimental design was applied to optimize the drying process by evaluating the effects of temperature, drying time, and air velocity on essential oil yield. The results showed that the three citrus species exhibited distinct physicochemical characteristics that influenced drying performance and extraction efficiency. Essential oil yield increased significantly with drying temperature, time, and air velocity, and the optimum conditions were identified as 70 ℃, 180 min, and 2 m s^{-1}. Biological evaluation revealed that essential oils extracted from fresh *Citrus limon* peels exhibited the strongest and broadest antimicrobial activity, whereas all drying treatments caused a noticeable reduction in antimicrobial effectiveness. Similarly, antioxidant activity, expressed as IC_{50} values, was highest in fresh samples and progressively decreased after open-air, convective, and infrared drying. Overall, the findings demonstrate that Tunisian citrus peels constitute a valuable source of bioactive essential oils and highlight the importance of selecting appropriate drying conditions to maximize extraction yield while minimizing losses in biological activity. These results provide useful guidance for the sustainable valorization of citrus processing by-products.

Keywords

Citrus Peels, Essential Oils, Drying Kinetics, Design of Experiments, Antimicrobial Activity, Antioxidant Activity, Waste Valorization

1. Introduction

The citrus processing industry generates substantial amounts of peel by-products, accounting for nearly 50% of the total fruit mass, which are often underutilized or discarded despite their high content of valuable bioactive compounds [1,

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2]. Citrus peels are particularly rich in essential oils, phenolics, and terpenoids, which exhibit well-documented antioxidant, antimicrobial, and functional properties, making them attractive for applications in food preservation, cosmetics, and nutraceuticals [1, 3, 4].

In the context of increasing environmental concerns and resource scarcity, the sustainable valorization of citrus residues has emerged as a key component of circular economy strategies aimed at minimizing waste while maximizing the recovery of high-value compounds [2, 5]. Recent research has emphasized the importance of developing integrated and energy-efficient processing routes that preserve the functional quality of citrus-derived bioactives while remaining economically viable at an industrial scale [5-7].

Among the processing steps involved in essential oil recovery, pretreatment operations—particularly drying—play a decisive role in determining extraction efficiency, chemical composition, and biological activity of the final product [3, 7]. Drying reduces moisture content, enhances storage stability, and facilitates oil release by altering peel microstructure and thermophysical properties [8, 9]. However, inappropriate drying conditions can negatively impact oil quality due to oxidative degradation, volatilization, or structural rearrangement of thermolabile compounds [3, 10].

Conventional thermal drying techniques such as hot-air and infrared drying are widely used due to their simplicity and scalability, yet they have been associated with significant losses of sensitive terpenoids and phenolics, resulting in reduced antioxidant and antimicrobial activities [11, 13]. Several studies have demonstrated that high temperatures and prolonged drying times accelerate oxidative reactions and promote the degradation of phenolic compounds and monoterpenes, thereby compromising the functional quality of citrus essential oils [3, 10, 11].

In contrast, mild drying strategies have gained increasing attention as a promising approach to balance oil yield, compositional integrity, and energy efficiency. Controlled drying conditions can enhance essential oil release by disrupting cellular structures while limiting oxidative losses and preserving thermolabile constituents [10, 14, 15]. Comparative assessments of drying techniques consistently confirm that process selection is critical for optimizing yield, chemical composition, and bioactivity of citrus peel essential oils [6, 7, 16].

Although limonene is typically the predominant compound in citrus essential oils, accumulating evidence indicates that minor terpenes such as α-terpinene, γ-terpinene, and other oxygenated monoterpenes contribute disproportionately to antioxidant and antimicrobial activities [6, 10, 17, 18]. The preservation of these minor constituents during drying is therefore essential for maintaining the functional quality and industrial value of citrus essential oils [6, 18]. Drying-induced compositional shifts can significantly alter the synergistic interactions among oil components, ultimately influencing bioactivity [11, 17].

Despite the growing body of literature on citrus peel valorization, limited studies have systematically examined the combined effects of drying methods on peel microstructure, essential oil composition, and biological activities, particularly for citrus varieties cultivated in North Africa. Tunisian citrus peels represent a largely unexplored resource with significant potential for sustainable industrial exploitation.

Therefore, the present study aims to evaluate the effects of different drying methods on the yield, chemical composition, and antioxidant and antimicrobial activities of essential oils extracted from Tunisian citrus peels. By integrating drying optimization, compositional profiling, and bioactivity assessment, this work seeks to provide scientifically grounded insights to support the development of sustainable and high-value industrial applications for citrus processing by-products.

2. Material and methods

2.1. Raw Material

Peels from three *Citrus* species were used: *Citrus sinensis* (E1, orange), *Citrus limon* (E2, lemon), and *Citrus aurantium* (E3, bitter orange). Samples were collected from the Nabeul region in northern Tunisia. After harvesting, peels were washed and carefully separated to obtain clean, homogeneous raw material. This preparation ensured freshness, geographical traceability, and removal of impurities, thereby facilitating subsequent analyses such as moisture content measurement, bulk density determination, thermo-physical analyses, drying kinetics, and essential oil extraction.

2.2. Physicochemical Characterization

2.2.1. Moisture Content

Moisture content of citrus peels was determined using infrared desiccation (IR moisture analyzer). Fresh peels were cut into uniform pieces, and 5–10 g of sample (initial mass, *Mi*) was placed in the desiccator at 100–105 ℃ until constant weight was achieved (30–60 min depending on sample size). Final mass (*Mf*) was recorded, and moisture content (*X*) was calculated as (1):

$$X(\%) = \frac{(M_i - M_f)}{M_f} \times 100 \quad (1)$$

This rapid and reliable infrared desiccation method enabled accurate determination of peel moisture content, a key parameter for raw material characterization and optimization of subsequent valorization processes.

Each assay was performed in triplicate for the three samples (E1, E2, E3), and mean values with standard deviations were reported.

2.2.2. Determination of Peel pH

The pH of fresh *C. sinensis*, *C. limon*, and *C. aurantium* peels was measured by homogenizing 1 g of peel in 10 mL of distilled water using a mortar and pestle. The suspension pH was determined with a calibrated pH meter. Measurements were performed in triplicate for each sample.

2.2.3. Determination of Soluble Sugar Content by Refractometry (Brix)

Soluble sugar content of citrus peels was assessed using a refractometer, which measures the refractive index of juice and expresses results in degrees Brix (°Bx), corresponding to the percentage of soluble solids. The instrument was calibrated prior to each series of measurements and cleaned between samples to prevent cross-contamination. This rapid method enables comparative evaluation of sugar content among samples and provides useful data for potential applications in food processing and by-product valorization.

2.2.4. Determination of Ash Content

Ash content of citrus peels was determined by dry ashing. Pre-weighed crucibles were sterilized at 600 ℃ for 30 min, cooled in a desiccator, and weighed (M_1). Fresh peel samples (2–3 g, cut into 0.5–1 cm pieces) were placed in the crucibles and weighed (M_2). Calcination was carried out in a muffle furnace at 600 ℃ for 4 h. After cooling in a desiccator, crucibles with ash were weighed (M_3). Ash content was calculated as (2):

$$T_c(\%) = \frac{(M_3 - M_1)}{(M_2 - M_1)} \times 100 \quad (2)$$

where M_1 = crucible mass (g), M_2 = crucible + sample before ashing (g), and M_3 = crucible + ash (g).

2.3. Physico-Thermal Characterization

2.3.1. Bulk Density

Bulk density (ρ), defined as mass per unit volume, was determined according to (3):

$$\rho = \frac{m}{V} \quad (3)$$

where m is the mass and V the volume. Sample volume was calculated using Archimedes' principle (4):

$$V = \frac{M_{air} - M_{water}}{\rho_{water}} \quad (4)$$

2.3.2. Porosity (ε)

Porosity (ε), expressed as the fraction of void volume relative to total volume, was calculated as (5):

$$\varepsilon = \frac{V_{pores}}{V_{total}} \times 100 \quad (5)$$

2.3.3. Volumetric Shrinkage

Drying generally increases bulk density, enhances porosity (water replaced by air), and induces progressive volumetric shrinkage.

For *C. sinensis*, *C. limon*, and *C. aurantium*, 6–7 peel fragments (~1 g each) were prepared. Initial mass (M_0) was measured using an analytical balance. Samples were dried in an infrared moisture analyzer at 70 ℃. At regular intervals (every 5–10 min), samples were removed for determination of mass in air (*Mair*) and in water (*Mwater*). After intermediate measurements, final drying at 100 ℃ for 10 min provided dry mass (*Ms*). Sample volume was calculated by (equation (4)).

Volumetric shrinkage (*RV*), representing volume reduction due to moisture loss during drying, was determined by (6):

$$RV = \frac{V_{initial} - V_{final}}{V_{initial}} \times 100 \quad (6)$$

2.3.4. Sorption Isotherms

Moisture sorption isotherms of citrus peels were determined by the static gravimetric method (Figure 1). Nine hermetically sealed glass jars, each containing sulfuric acid solutions of different concentrations, provided controlled relative humidity environments. Perforated aluminum molds containing 3 g peel samples (*C. sinensis*, *C. limon*, *C. aurantium*) were suspended above the solutions.

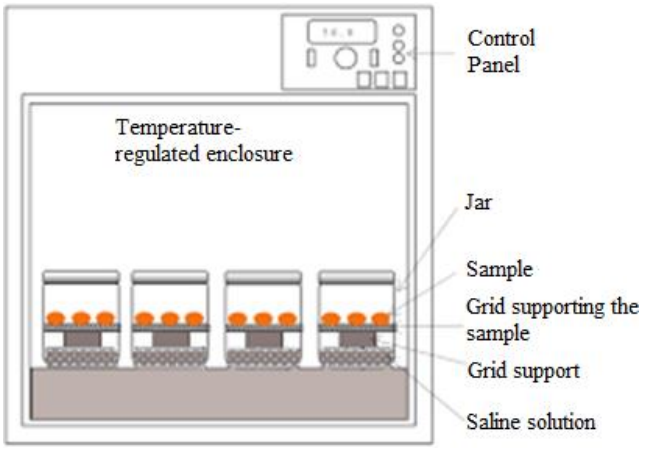


Figure 1. *Static gravimetric method.*

For adsorption studies, jars were stored at 50, 60, and 70 ℃ (one week per condition). Desorption was evaluated by decreasing the temperature stepwise (70 → 60 → 50 ℃), with weekly weighing. Dry mass of samples was determined at 105 ℃ for 10 min using an infrared desiccator.

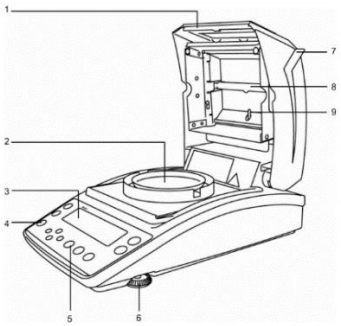
This method yielded adsorption–desorption isotherms, providing insights into the hygroscopic behavior of citrus peels, critical for storage optimization and valorization processes.

2.4. Drying Kinetics

Drying kinetics were investigated for *C. sinensis*, *C. limon*, and *C. aurantium* peels using three methods: infrared drying, forced-convection oven drying, and open-air drying.

2.4.1. Infrared Drying

Infrared drying was performed using a moisture analyzer equipped with a sample tray and a digital control panel (Figure 2). The instrument allowed regulation of temperature and time, and provided real-time data on drying duration, residual mass percentage, and sample weight. Drying was conducted either under fixed-time mode (10 min intervals) or automatic mode, in which the process terminated upon attainment of constant mass ("test over" displayed).



Pos.	Description
1	Fenêtre de regard
2	Cuvette porte-échantillon
3	Afficheur
4	Bulle d'air
5	Clavier
6	Pied d'ajustage
7	Hotte chauffante
8	Lampe halogène
9	Capteur de température

Figure 2. *Analyseur d'humidité IR.*

For each species, 5 g of peel were placed in the infrared moisture analyzer and dried at 50, 60, and 70 ℃ in AUTO mode. Sample mass was recorded every 3 min until the instrument stopped automatically ("test over"). Dry mass was then determined at 105 ℃ for 10 min. Each experiment was performed in triplicate.

2.4.2. Forced-Convection Drying

In the forced-convection oven (Figure 3), 5 g peel samples were placed on a balance-equipped support introduced through a plexiglass window. After tare adjustment, drying was performed at 50, 60, or 70 ℃ with air velocity set to ~1 m/s (laminar flow, verified with an anemometer). Sample mass was recorded every 5 min during the first hour and every 15 min thereafter, requiring temporary interruption of airflow to avoid measurement disturbance. Drying was continued (3–7 h depending on species and temperature) until constant mass was reached. Final dry mass was determined with the infrared analyzer at 105 ℃ for 10 min.

The collected data were used to construct drying curves, illustrating moisture loss and drying kinetics for each citrus species and drying method.

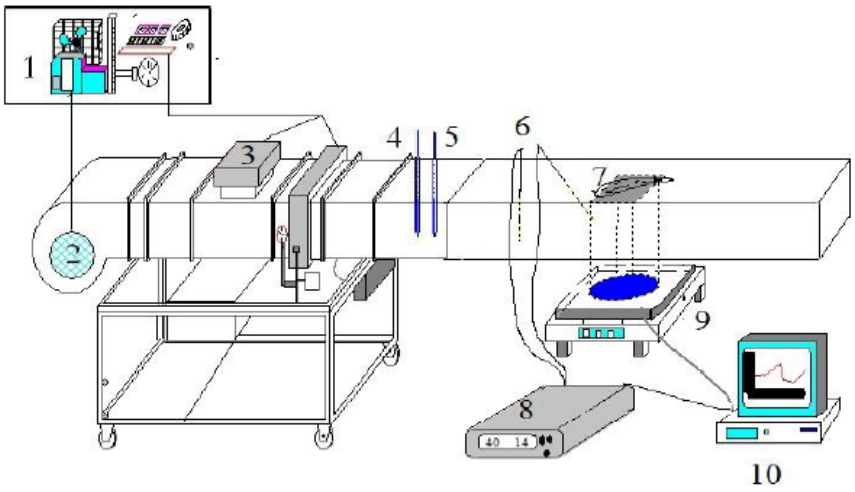


Figure 3. *Schematic representation of the drying tunnel.*

(1) electrical cabinet, (2) fan, (3) resistance block, (4) thermo-hygrometer, (5) anemometer, (6) thermocouples, (7) sample, (8) data acquisition system, (9) balance, (10) computer.

Thin-layer forced-convection drying of citrus peels (*Citrus sinensis*, *Citrus limon*, and *Citrus aurantium*) was performed under controlled thermal and aeraulic conditions. Air velocity in the drying chamber was monitored using a vane anemometer (Testo 435-3, accuracy ±0.1 m/s), while temperature and relative humidity at the inlet were measured with a thermo-hygrometer (Testo 635-2, accuracy ±2.5 ℃ and ±1% RH). To ensure stable drying conditions, the airflow system was operated for at least one hour before loading the samples.

Peels were cut into approximately 5 g portions and spread in a thin layer on a perforated tray, which was placed in the drying chamber on an electronic balance (Kern 440-35A, capacity 200 g, accuracy ±0.01 g). The balance was connected to a computer for continuous mass monitoring.

2.4.3. Open-air Drying

After cleaning, peeling, and cutting into small pieces, samples from the three citrus varieties were shade-dried for 3–4 days. The dried peels were then ground and prepared for essential oil extraction.

2.5. Optimization Using Design of Experiments (DoE)

Design of Experiments (DoE) is a statistical method that defines a series of trials based on an optimal strategy, enabling the prediction of a response with minimal error and the least number of experiments, according to a proposed model. DoE involves selecting and organizing experiments to identify, at low cost, the effects of process parameters on product responses. These methods rely on straightforward mathematical and statistical concepts.

The modeling and study of the effects of drying parameters on the essential oil yield of selected citrus peels were carried out using DoE with NEMRODW software.

A full factorial 2^3 design was selected, including three factors (Temperature, Drying Time, and Air Velocity), each studied at two levels (low and high). Table 1 below presents the factors and their corresponding experimental levels.

Table 1. *Factors and Experimental Domain.*

Factor	Low Level (-1)	High Level (+1)
Temperature (℃)	50	70
Drying Time (h)	30	180
Air Velocity (m/s)	1	2

The low and high levels of the factors were carefully selected to focus exclusively on the falling-rate drying period and to ensure operation under laminar flow conditions.

The relationship between the response and the factors (variables) was described using a first-order polynomial model (7): First-order polynomial modeling of the response as a function of the variables.

$$Y = \beta_0 + \sum_{i=1}^{k} \beta_i X_i \quad (7)$$

Where:

Y is the predicted response (e.g., essential oil yield),

β_0 is the intercept,

β_i are the regression coefficients representing the effect of each factor,

X_i are the coded values of the independent variables (factors),

k is the number of factors studied.

This linear model allows estimating the main effects of each factor on the response and provides a basis for identifying optimal drying conditions.

The experimental matrix and the design of experiments for this study are presented in Table 2.

Table 2. *2^3 factorial design for the optimization of essential oil extraction from citrus peels.*

N °	Variables naturelles			Variables cod ées			
	T	Temps	Vitesse	X_1	X_2	X_3	X_1 X_2 X_3
1	50	30	1	-1	-1	-1	-1

N °	Variables naturelles			Variables codées			
	T	Temps	Vitesse	X_1	X_2	X_3	X_1 X_2 X_3
2	70	30	1	+1	-1	-1	+1
3	50	180	1	-1	+1	-1	+1
4	70	180	1	+1	+1	-1	-1
5	50	30	2	-1	-1	+1	+1
6	70	30	2	+1	-1	+1	-1
7	50	180	2	-1	+1	+1	-1
8	70	180	2	+1	+1	+1	+1

This table summarizes all combinations of the three factors—temperature, drying time, and air velocity—at their low and high levels, allowing for the evaluation of their individual and combined effects on the response variable.

2.6. Essential Oil Extraction

Essential oils were extracted from fresh or dried peels of *Citrus sinensis* (E1), *Citrus limon* (E2), and *Citrus aurantium* (E3). Extractions were performed using a steam distillation apparatus.

Essential oils were obtained using a Clevenger-type apparatus (Figure 4). Briefly, the desired amount of peel was weighed and placed in a glass flask filled one-third with distilled water and one-third with plant material, leaving the remaining third empty to allow boiling. The extraction setup, including the flask, Clevenger apparatus, condenser, and water inlet/outlet tubing, was assembled.

The flask was heated to 70 ℃ until water boiled, then maintained at 50 ℃. Oil yield was monitored every 15 min using the Clevenger graduation. Extraction was stopped when the yield reached a plateau, typically after 3–4 h.

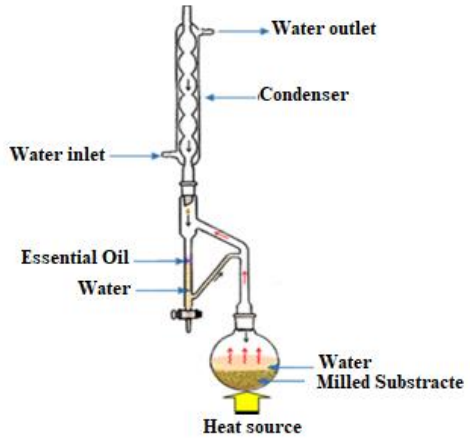


Figure 4. *Essential Oil Extraction Using a Clevenger Apparatus.*

The oil yield (%) was calculated as (8):

$$Yield \quad (\%) = \frac{m_{oil}}{m_{peel}} \times 100 \tag{8}$$

where m_{oil} is the mass of extracted oil and m_{peel} is the initial peel mass.

The extracted essential oils were subsequently used for all analyses and assays reported in this study.

2.7. Chemical Composition by GC-MS

The chemical composition of the essential oils (EOs) was analyzed using gas chromatography coupled with mass spectrometry (GC-MS). Analyses were performed on an Agilent 7890A GC coupled to an Agilent 5975C inert XL MSD (electron ionization, 70 eV) equipped with an HP-5MS capillary column (30 m × 0.25 mm, 0.25 μm film). The column temperature program was: 50 ℃ for 2 min, ramped to 110 ℃ at 8 ℃/min, held for 8 min, then ramped to 210 ℃ at 10 ℃/min for 3 min, and finally to 300 ℃ at 12 ℃/min. The transfer line was maintained at 250 ℃. Helium served as the carrier gas at 1 mL/min with a 100:1 split ratio. Mass spectra were scanned from 50–550 m/z at 1 s/scan. Compound identification was based on comparison with the Wiley 09 and NIST 2011 mass spectral libraries.

2.8. Antimicrobial Activity

Antimicrobial activity was evaluated against human pathogenic bacteria and yeast:

1) *Reference strains: Staphylococcus aureus* ATCC 29213, *Pseudomonas aeruginosa* ATCC 27853, *Enterococcus faecalis* ATCC 29212, *Listeria monocytogenes* ATCC 19115, *Escherichia coli* ATCC 35218.
2) *Clinical isolates: Salmonella arizonae* DMB 560, *Klebsiella pneumoniae*, methicillin-resistant *S. aureus* (MRSA).
3) *Fungal strain: Candida albicans* ATCC 10231.

Bacteria were cultured on Luria Broth (LB) and *C. albicans* on Winge Broth (WB) agar at 30 ℃ for 24 h. Pre-cultures were suspended in saline to 10^7 CFU/mL and spread on Mueller-Hinton agar. Wells (6 mm diameter) were loaded with 10 µL of essential oil. Inhibition zones were measured after incubation; tests were performed in triplicate.

2.9. Antioxidant Activity

The radical scavenging activity of essential oils was assessed using the DPPH assay. Briefly, 100 µL of EO at varying concentrations was mixed with 100 µL of 6 × 10^{-5} M DPPH solution, incubated in the dark for 30 min, and absorbance measured at 517 nm (BioTek ELx80). Radical scavenging was calculated as (9):

$$Inhibition\ \ (\%) = \frac{A_{control} - A_{sample}}{A_{control}} \times 100 \qquad (9)$$

The IC_50 (concentration producing 50% inhibition) was determined from the dose–response curve; lower IC_50 values indicate higher antioxidant activity. All measurements were performed in triplicate.

3. Results and Discussion

3.1. Physicochemical Analyses

Physicochemical analyses showed high moisture contents (70–76%) and acidic pH values (3.8–4.5) for all citrus peels. Thermophysical characterization revealed species-specific differences in porosity and shrinkage, with *C. aurantium* exhibiting the most stable structure during drying (Table 3).

Table 3. *Physicochemical characteristics of citrus peels.*

	E1 *Citrus sinensis*	**E2 *Citrus limon***	**E3 *Citrus aurantium***
Water content *X* (kg.kg^{-1} dry material)	0.73 ±0.77	0.76 ±0.64	0.71 ±0.48
pH	4.52 ±0.04	3.82 ±0.03	4.22 ±0.03
Soluble Sugar Content (°Brix)	10.40 ±0.20	7.80 ±0.21	9.50 ±0.20
Ash Content (%)	7.51 ±0.05	8.02 ±0.14	5.67 ±0.23

3.1.1. Moisture Content

The moisture content of citrus peels was determined by infrared desiccation and expressed as mean values from triplicate measurements (Table 4). Average values were 72.83 ± 0.77% for *Citrus sinensis*, 75.77 ±0.64% for *Citrus limon*, and 70.32 ± 0.48% for *Citrus aurantium*. These results are consistent with those reported in the literature, e.g., 73.2% for *C. sinensis* peels from Tunisia [19], 76.4% for *C. limon* [20], and 69.8% for *C. aurantium* from Greece [21]. The relatively high water content (70–76%) highlights the need to consider drying and storage conditions prior to downstream valorization and extraction processes.

3.1.2. PH

The pH of citrus peel matrices is a critical factor influencing their bioactive properties, particularly antioxidant activity. In our study (Table 4), *C. limon* peels exhibited the lowest pH (3.82 ±0.03), followed by *C. aurantium* (4.22 ±0.03) and *C. sinensis* (4.52 ± 0.04), reflecting differences in organic acid content. The acidic environment of citrus peels can enhance the stability and solubility of phenolic compounds, flavonoids, and ascorbic acid, which are major contributors to antioxidant activity. Previous studies have reported a direct correlation between lower pH and higher antioxidant potential in citrus peel extracts, as the acidic conditions prevent oxidation and degradation of bioactive compounds during extraction and storage [22, 23]. This relationship suggests that species with lower peel pH, such as *C. limon*, may offer greater functional benefits when used in nutraceuticals, food additives, or natural antioxidants in industrial applications. Understanding the interplay between pH and antioxidant activity is therefore essential for optimizing the valorization of citrus by-products and improving their efficacy in food and pharmaceutical formulations.

3.1.3. Soluble Solids (°Brix)

The average soluble solid content (SSC) of fresh citrus peels varied among the three species studied, with *C. sinensis* exhibiting the highest SSC (10.4 ±0.2 °Brix), followed by *C. aurantium* (9.5 ± 0.2 °Brix) and *C. limon* (7.8 ± 0.2 °Brix). Soluble solids, primarily composed of sugars, organic acids, and other soluble compounds, are important indicators of fruit maturity and quality, and they can influence both taste and functional properties of citrus peel extracts. The observed differences in SSC are consistent with previous reports indicating

higher sugar accumulation in sweet orange peels compared to lemon and bitter orange peels [22, 23]. The higher SSC in *C. sinensis* may also contribute to enhanced palatability and potential use in food applications, whereas the lower SSC in *C. limon* aligns with its more acidic profile, reflecting the inverse relationship often observed between sugar content and acidity in citrus matrices. These findings provide useful reference values for the physicochemical characterization and valorization of citrus by-products in industrial applications.

3.1.4. Ash Content

The ash content of fresh citrus peels, reflecting the total mineral composition, varied significantly among the three species. *C. limon* exhibited the highest ash content (8.02%), followed by *C. sinensis* (7.51%), while *C. aurantium* had the lowest value (5.67%). These differences may be attributed to species-specific variations in mineral accumulation, environmental growth conditions, and peel tissue composition. The relatively high ash content in *C. limon* peels suggests a greater concentration of essential minerals, which could enhance their nutritional and functional potential in food and industrial applications [22]. In contrast, the lower ash content of *C. aurantium* indicates a comparatively reduced mineral load, which may influence its utility in mineral-enriched products. These findings are consistent with previous reports highlighting species-dependent variations in the mineral profiles of citrus by-products and underscore the importance of mineral content in evaluating the functional properties of citrus peels.

3.2. Physicothermal Characterization

3.2.1. Bulk Density, Porosity, and Volumetric Shrinkage

Figure 5 shows the variation of bulk density of citrus peels (*C. sinensis*, *C. limon*, *C. aurantium*) as a function of moisture content. These measurements provide key insights into the structural stability of the peels during drying and are essential for understanding mass and heat transfer phenomena, as well as predicting textural changes throughout dehydration.

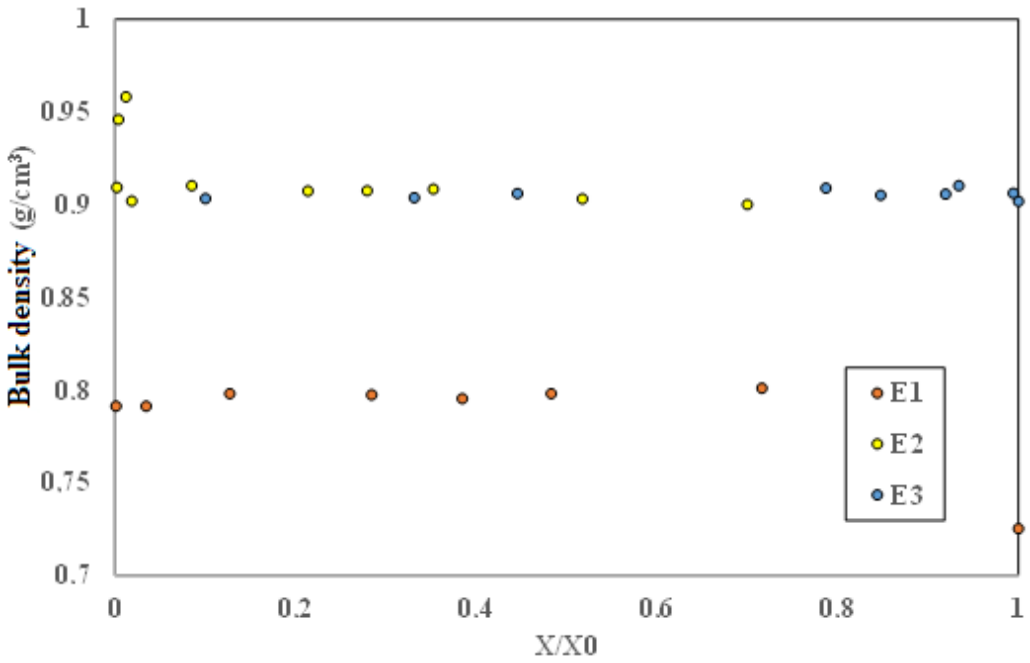


Figure 5. *Bulk density versus moisture content for the three citrus peel species.*

The results indicate relatively stable bulk densities across the full moisture range, with *C. aurantium* (E3) exhibiting the highest values, followed by *C. limon* (E2) and *C. sinensis* (E1). The limited variation in bulk density with moisture suggests a stable cellular structure during dehydration.

As shown in Figure 6, volumetric shrinkage increased progressively with decreasing moisture content, highlighting interspecific differences in structural response to water loss. These variations reflect distinct textural and morphological properties of the peels, which may influence their drying kinetics and potential applications.

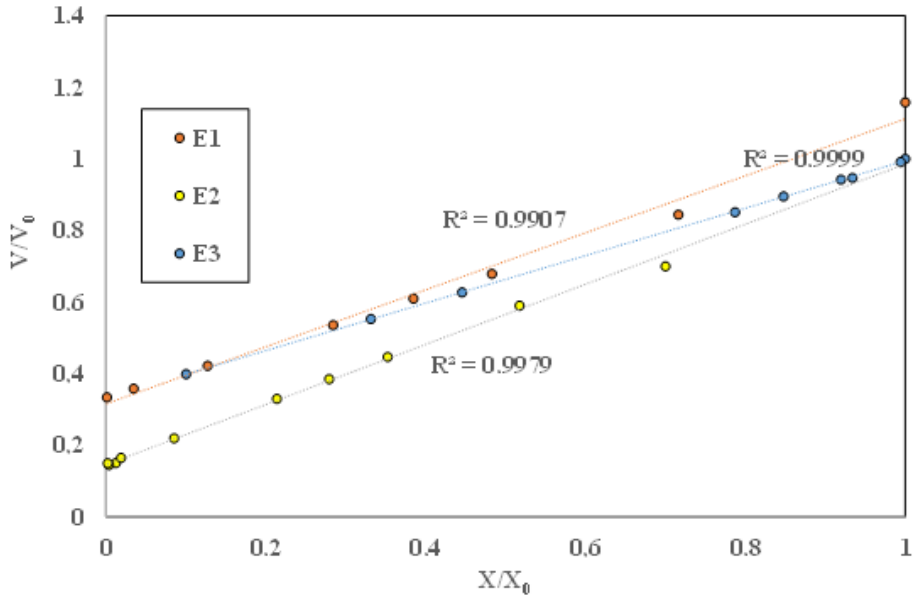


Figure 6. *Volumetric shrinkage.*

All species exhibited a linear relationship between volumetric shrinkage and moisture content. *C. sinensis* (E1) showed the highest shrinkage, followed by *C. limon* (E2) and *C. aurantium* (E3). The high coefficients of determination (R^2) confirm the strong correlation between water loss and volume reduction.

Figure 7 depicts the evolution of peel porosity with decreasing moisture content. The results highlight progressive changes in internal structure during dehydration, reflecting interspecific differences in tissue organization that may affect mass transfer and drying behavior.

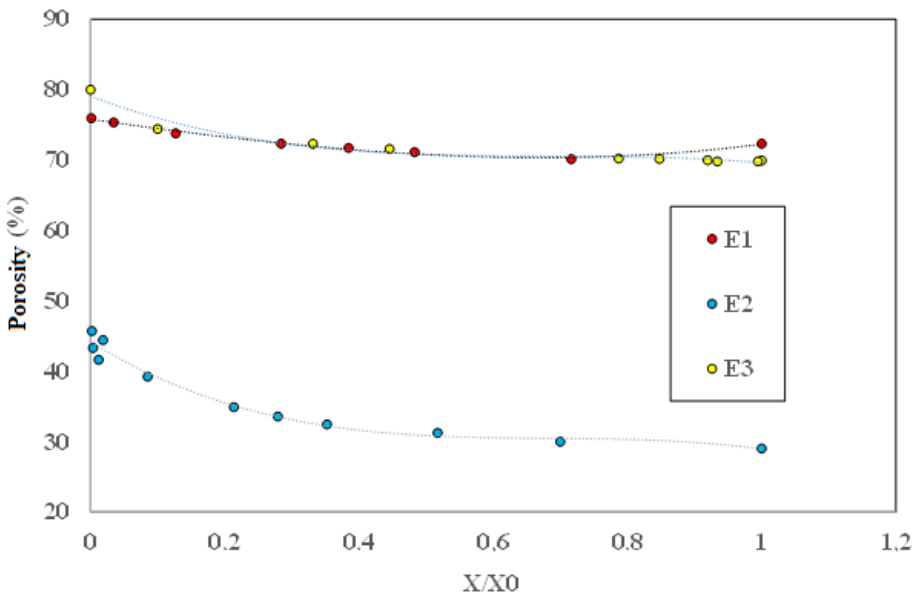


Figure 7. *Porosity evolution.*

During drying, *C. aurantium* (E3) exhibited the highest porosity, which remained relatively stable throughout the process (Figure 7). In contrast, *C. sinensis* (E1) and *C. limon* (E2) showed significant reductions in porosity, with E2 displaying the most pronounced decrease.

These results showed that the three citrus species displayed distinct physical characteristics in bulk density and porosity, reflecting variations in cellular structure and composition.

Volumetric shrinkage was linearly correlated with moisture content for all species, but with different magnitudes. Porosity evolution was species-dependent, with E3 maintaining the highest stability.

C. *sinensis* (E1), with its higher shrinkage, may require closer control of drying conditions. C. *aurantium* (E3) preserved its porous structure more effectively, potentially facilitating moisture removal and essential oil extraction. *C. limon* (E2) exhibited intermediate behavior but with a marked porosity reduction that could impact final product properties.

Species-specific drying conditions may be necessary to preserve structural integrity. The relative stability of bulk density indicates that volume changes were primarily water-driven, which is favorable for product quality retention.

3.2.2. Sorption Isotherms

Figure 8 depicts the desorption isotherms of orange peels (*C. sinensis*, E1) at three temperatures (50, 60, and 70 ℃). The curves describe the relationship between ambient relative humidity and equilibrium moisture content, highlighting how water retention capacity decreases with increasing temperature and varies according to environmental conditions.

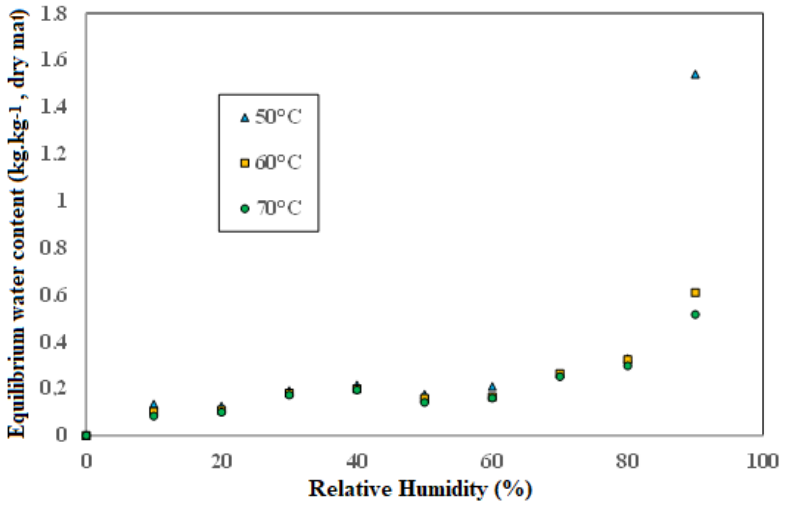


Figure 8. *Desorption isotherms of orange peels (E1).*

The isotherms (Figure 8) show that equilibrium moisture content increases with relative humidity. A clear temperature effect is observed: at a given relative humidity, equilibrium moisture content decreases as temperature rises. The curves become steeper above 80% relative humidity, indicating accelerated water uptake under high-humidity conditions.

Figure 9 presents the desorption isotherms of lemon peels at the same temperatures as orange. Comparison reveals distinct hygroscopic behaviors between the two species, reflecting differences in tissue structure and chemical composition.

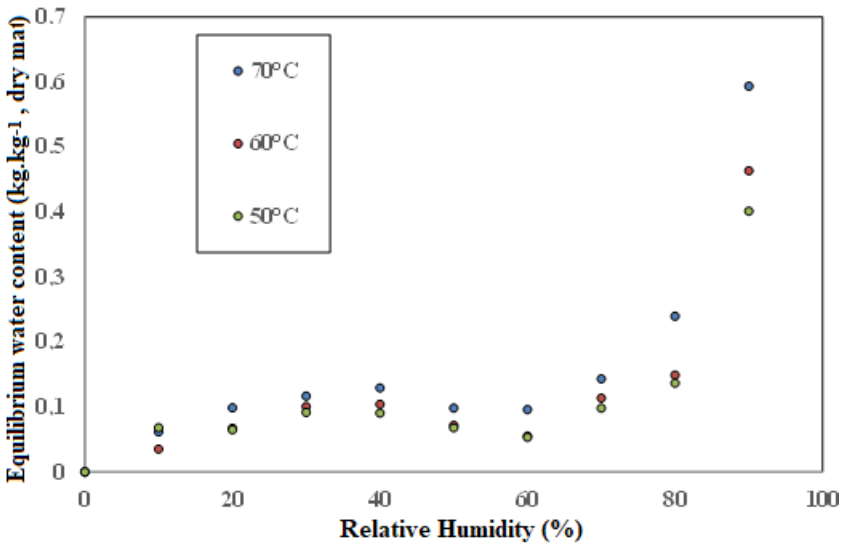


Figure 9. *Desorption isotherms of lemon peels (E2).*

The isotherms (Figure 9) follow similar trends to orange (E1), but with generally lower equilibrium moisture contents. The temperature effect is less pronounced, particularly at intermediate relative humidities. A sharp increase in equilibrium moisture is observed under very high relative humidity (>80%).

Figure 10 presents the desorption isotherms of bitter orange peels. Comparison with E1 and E2 highlights interspecific differences in water retention behavior under varying temperature and humidity conditions.

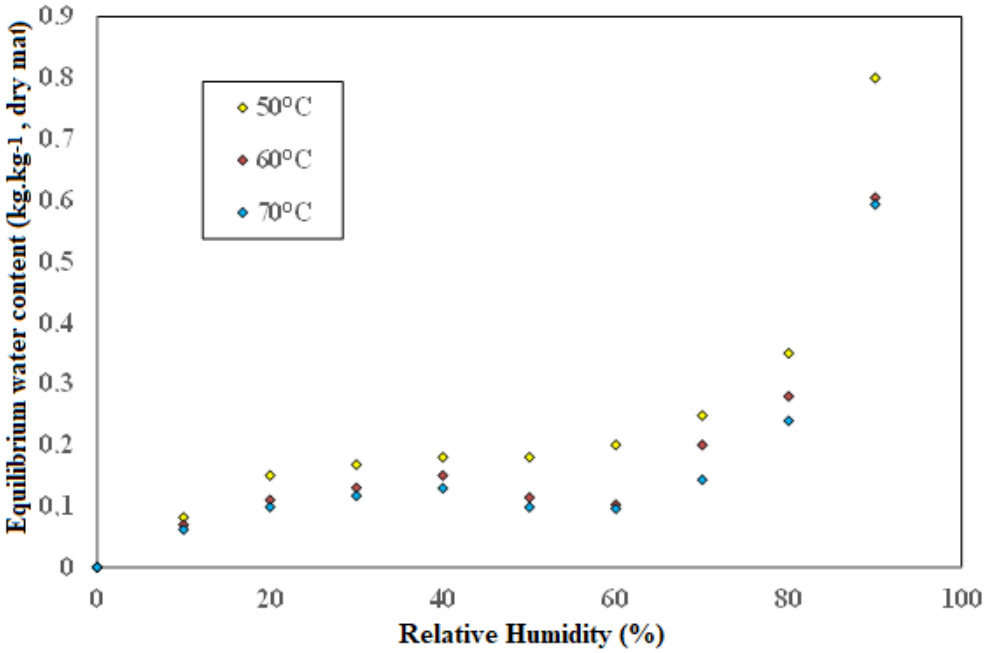


Figure 10. *Desorption isotherms of bitter orange peels (E3).*

The desorption curves of E3 follow the general trend observed for E1 and E2. The effect of temperature is more pronounced, particularly at high relative humidity. Equilibrium moisture contents reach higher values than those of E1 and E2 under high humidity conditions.

Figure 11 presents both adsorption and desorption isotherms for E3 at 50 ℃, highlighting the phenomenon of hysteresis. This effect reflects the molecular relaxation occurring between adsorption and desorption, indicating that water uptake and release do not follow identical paths.

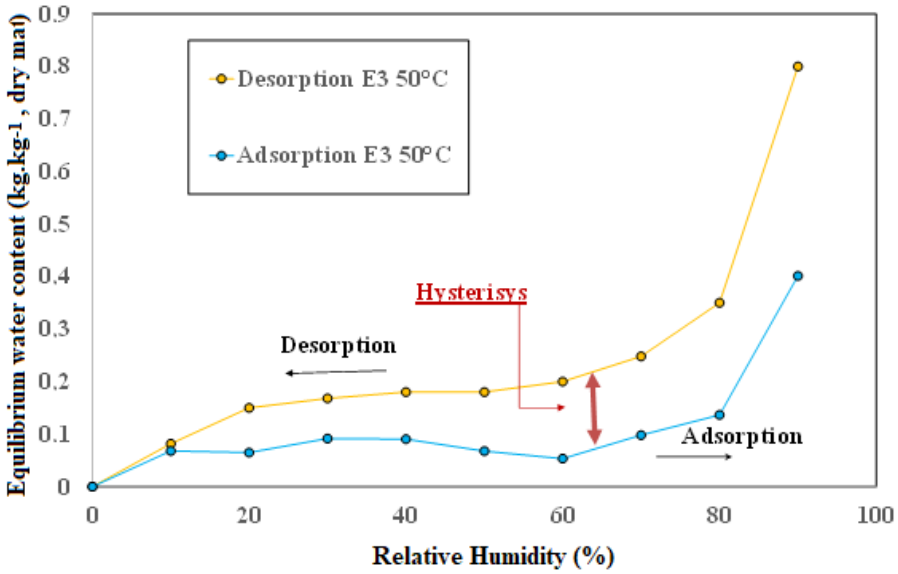


Figure 11. *Desorption and Adsorption Isotherms of Bitter Orange (E3) – Hysteresis.*

The desorption curve lies above the adsorption curve, indicating that the peels retain more water during drying than during rehydration. The hysteresis gap is most pronounced at intermediate relative humidities.

Equilibrium moisture content of citrus peels increased with relative humidity for all species, with bitter orange (E3) showing the highest values, particularly at high humidity, while lemon (E2) exhibited generally lower values. Temperature inversely affected moisture content, with higher temperatures reducing water retention at a given humidity. Hysteresis was observed in E3, with the desorption curve lying above the adsorption curve, indicating greater water retention during drying than rehydration. Differences among species likely reflect variations in cellular structure and chemical composition, influencing water absorption and retention. These findings highlight the need to tailor drying and storage conditions to each species to preserve structural integrity and optimize processing efficiency.

3.3. Drying Kinetics

Drying kinetics were strongly influenced by temperature and method. Infrared drying provided faster moisture removal, whereas convective drying resulted in smoother and more uniform drying curves.

Figure 12 shows the corresponding drying rates versus reduced moisture content, confirming faster kinetics at elevated temperatures.

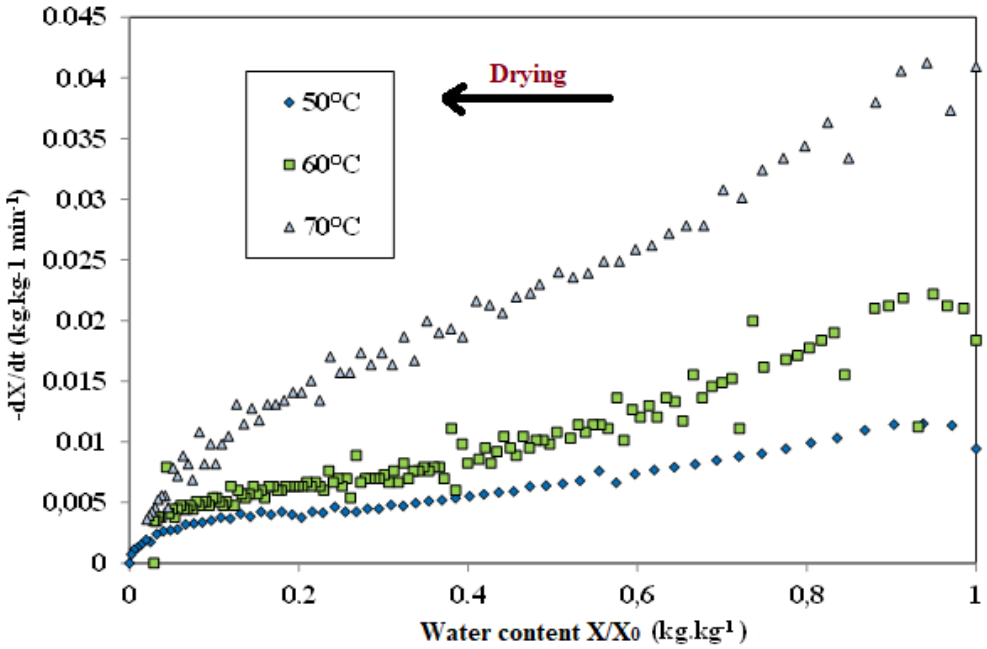


Figure 12. *Drying Rate of E2 by IR.*

For all temperatures, the drying rate increased as moisture content decreased, an unusual behavior that may reflect specific material properties or the IR drying method. The drying rate was highest at 70 ℃, followed by 60 ℃ and 50 ℃, consistent with observed moisture loss trends. Fluctuations were observed, particularly at 60 ℃ and 70 ℃, likely due to the heterogeneous structure of the peel or minor variations in drying conditions. No distinct constant-rate period was observed, as is typical for many biological materials. The increasing drying rate at lower moisture contents may be linked to structural changes in the citrus peel during dehydration.

Figure 13 shows the evolution of reduced moisture content of orange peels (E1, *Citrus sinensis*) over time at 50 ℃, 60 ℃, and 70 ℃ under forced convection drying.

The drying rate clearly increased with temperature. At 70 ℃, the peel reached near-zero moisture content in approximately 195 minutes, compared with 300 minutes at 60 ℃ and over 300 minutes at 50 ℃. Two main drying phases were observed: an initial rapid drying phase (up to ~50–60 minutes) followed by a slower drying phase. The transition between these phases was more pronounced at 70 ℃ than at lower temperatures. The drying curves (Figures 12 and 13) show an initial fast water loss, followed by a gradual slowdown, reflecting the typical pattern where free water evaporates quickly before the slower removal of bound water.

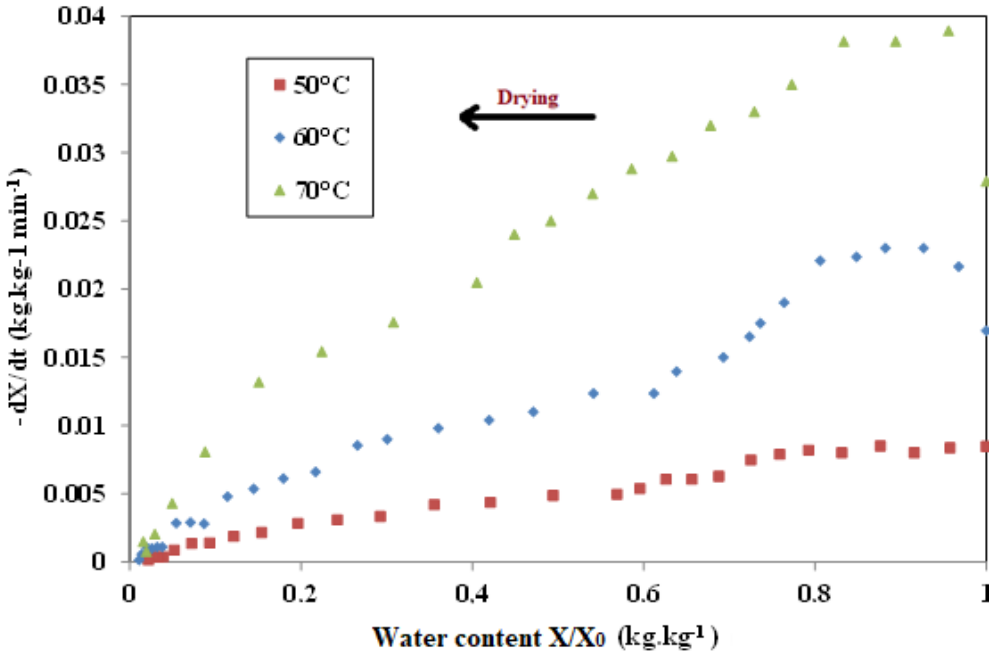


Figure 13. *Convective Drying Rate of E1.*

Temperature strongly influenced the drying rate. At 70 ℃, moisture content decreased more rapidly than at 60 ℃ or 50 ℃. Slower drying at lower temperatures, however, may better preserve bioactive compounds in the peels, despite requiring longer processing times.

Understanding the drying kinetics at different temperatures allows selection of optimal conditions depending on the goal—speed versus extract quality. For rapid and efficient drying, 70 ℃ appears ideal, whereas 60 ℃ or 50 ℃ may be preferable when preserving bioactive properties. The observed increase in drying rate as moisture content decreases may reflect structural changes in the peel or improved heat penetration as drying progresses.

Figure 14 compares convective and IR drying at 70 ℃ for samples E2 and E3, highlighting differences in drying efficiency and kinetics between the two methods.

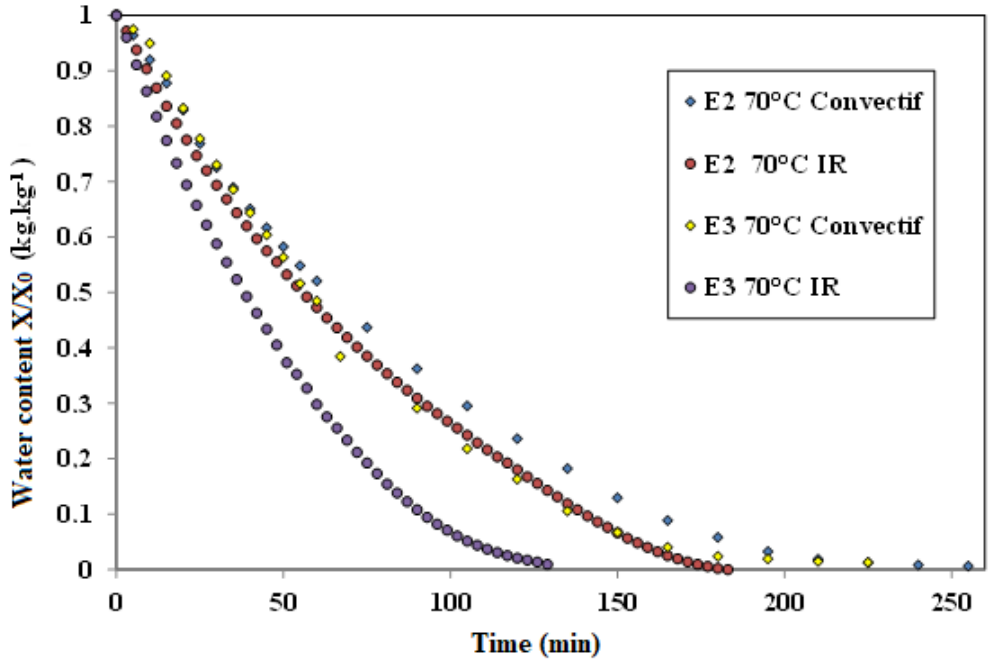


Figure 14. *Effect of Drying Method on Moisture Loss.*

Drying kinetics of citrus peels were strongly influenced by temperature and drying method. For IR drying of E2, moisture content decreased exponentially over time, with higher temperatures accelerating drying; 70 ℃ reached near-zero moisture in ~180 min, whereas 50 ℃ required over 400 min. Interestingly, the drying rate increased as moisture decreased, suggesting structural changes in the peel or enhanced heat penetration. Convective drying of E1 showed a typical two-phase pattern: a rapid initial moisture loss followed by slower removal of bound water, with higher temperatures (70 ℃) achieving faster drying than 60 ℃ or 50 ℃. While rapid drying favors processing efficiency, lower temperatures better preserve bioactive compounds. Comparison between IR and convective drying at 70 ℃ for E2 and E3 revealed method-dependent differences in drying kinetics, highlighting the need to balance drying speed and product quality. Overall, temperature and method selection are critical for optimizing peel drying while maintaining structural integrity and functional properties.

For E2 (*Citrus limon*), convective and IR drying showed comparable performance, with IR slightly faster toward the end of the process. In contrast, E3 (*Citrus aurantium*) dried significantly faster under IR than convective conditions. Regardless of the method, E3 dried more rapidly than E2. All drying curves exhibited typical exponential decay. The relative efficiency of IR versus convective drying was species-dependent, with E3 more sensitive to the drying method than E2. These results suggest that the choice of drying method should be optimized according to the specific citrus species.

Overall, trends were similar between convective drying of E1 and IR drying of E2; however, convective drying produced smoother drying rate curves, indicating a more uniform process.

3.4. Optimization Via Experimental Design DoE

Optimization via DoE demonstrated that temperature was the most influential factor on essential oil yield, followed by drying time and air velocity (Table 3).

The experimental matrix for this study, showing responses in terms of essential oil yield for the three citrus varieties, is presented in Table 4.

Table 4. *Experimental Matrix.*

N °	Natural Variables			Coded Variables			Responses: Essential Oil Yields		
	Temperature (°C)	Time (min)	Air velocity (m/s)	X_1	X_2	X_3	Y_{E1} (%)	Y_{E2} (%)	Y_{E3} (%)
1	50	30	1	-1	-1	-1	0.43	0.14	0.20
2	70	30	1	+1	-1	-1	0.27	0.15	0.21
3	50	180	1	-1	+1	-1	0.45	0.39	0.38
4	70	180	1	+1	+1	-1	0.55	0.31	0.28
5	50	30	2	-1	-1	+1	0.25	0.24	0.19
6	70	30	2	+1	-1	+1	0.27	0.16	0.25
7	50	180	2	-1	+1	+1	0.57	0.35	0.55
8	70	180	2	+1	+1	+1	0.69	0.31	0.58

The analysis of all interaction effects for orange peel (E1) is summarized in Figure 15. Interaction effects for the other two varieties (lemon and bitter orange) are not presented in this study.

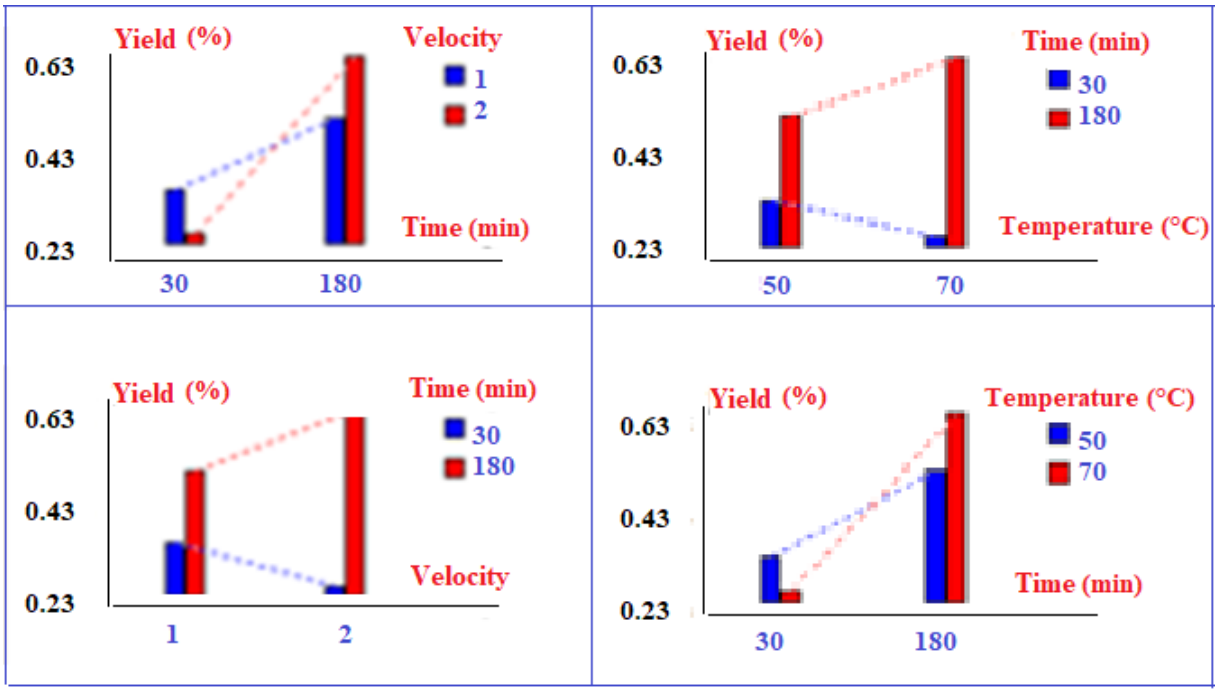


Figure 15. Interaction Effects.

At an air velocity of 1 m/s, the essential oil yield increases significantly with drying time, rising from 0.23 to approximately 0.63. At 2 m/s, yield also improves with time, with overall performance higher than at 1 m/s. The curves indicate a significant interaction between drying time and air velocity: longer drying at higher air velocity produces greater yields. Specifically, increasing air velocity further enhances yield at 180 minutes of drying. The steep positive slopes highlight that air velocity has a pronounced effect on yield during extended drying periods. Overall, interactions between temperature, drying time, and air velocity significantly influence essential oil yield from orange peel, with higher temperatures and longer drying times, particularly at higher air velocities, resulting in markedly increased yields.

Equation (10) provides a predictive model for estimating the essential oil yield from orange peel under different convective drying conditions, incorporating the effects of temperature, drying time, and air velocity.

$$\hat{Y} = 13.57 + 0.749X_1 + 4.277X_2 + 0.434X_3 + 0.003X_1X_2 + 0.134X_1X_3 - 0.059X_2X_3 \quad (10)$$

The ANOVA results (Table 5) confirm the reliability of the predictive model. The regression shows a significance level of 95.5%, while the model validity reaches 90.5%, both indicating strong statistical robustness. Experimental yields were estimated using the mathematical model described in equation (10) (Figure 16). A close agreement between experimental and predicted values was observed, with a high regression coefficient ($R^2 = 0.9827$), confirming the model's accuracy and predictive power.

Table 5. Analysis of variance (ANOVA) for the predictive model.

Source of variation	Sum of squares	df	Mean square	F-ratio	Significance (%)
Regression	1.47×10^2	4	3.56×10^1	0.15	95.5
Residuals	1.05×10^4	45	2.31×10^2	–	–
Validity	1.57×10^3	12	1.31×10^2	0.49	90.5
Error	9.06×10^3	34	2.55×10^2	–	–
Total	1.07×10^4	50	–	–	–

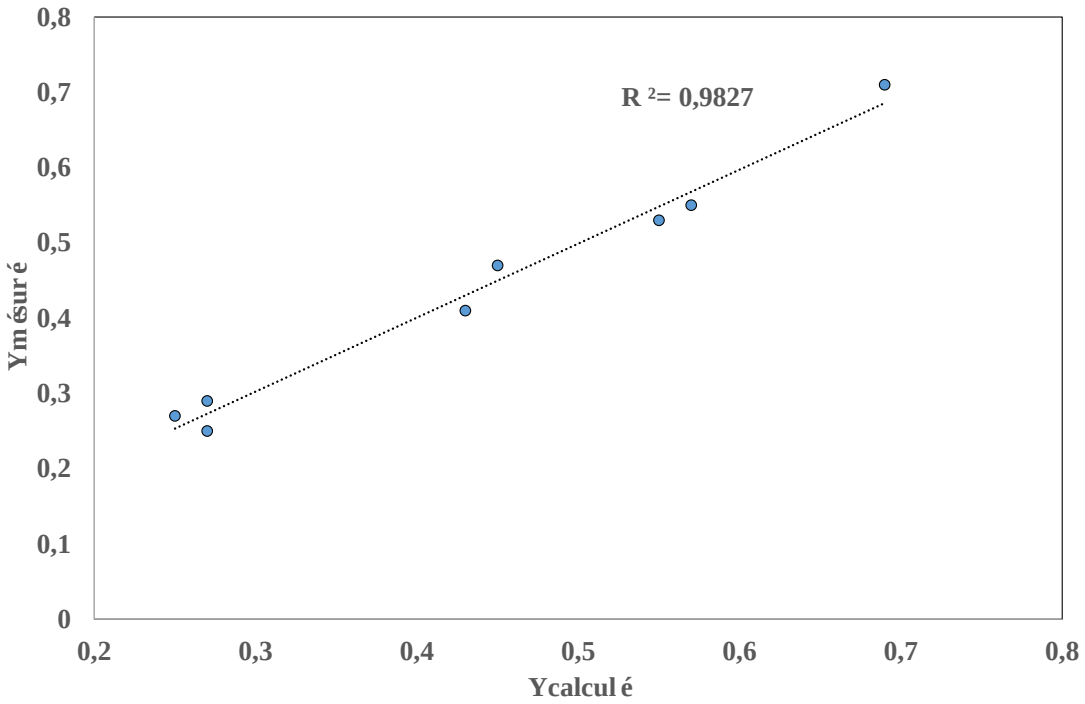
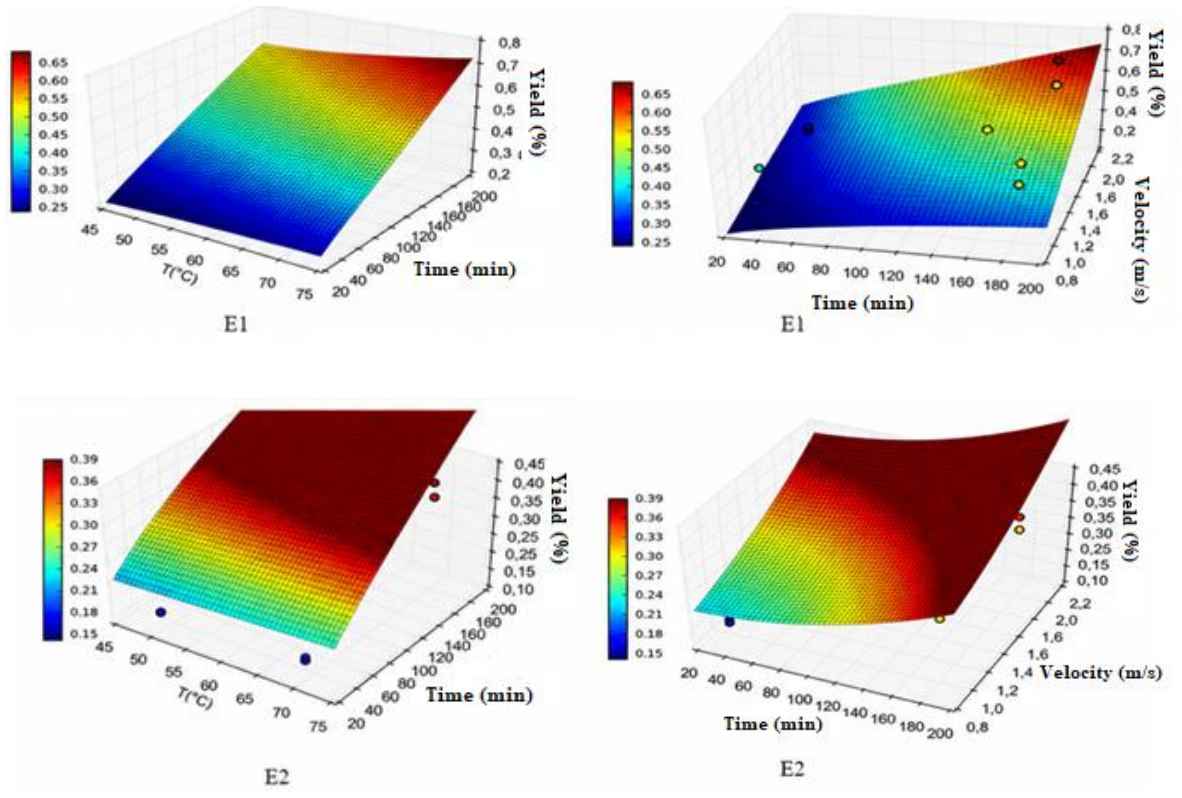


Figure 16. *Measured versus predicted yields of essential oils from orange peels (E1) using the mathematical model.*

The strong correlation (R ²= 0.9827) confirms the accuracy and predictive reliability of the model.

The yield model was used to generate the corresponding response surface, analyzed with the software *Curv-Expert Professional* (Figure 17). The analysis confirms that optimal essential oil yield can be achieved within the studied experimental domain, indicating the relevance of the chosen operating conditions for process optimization.



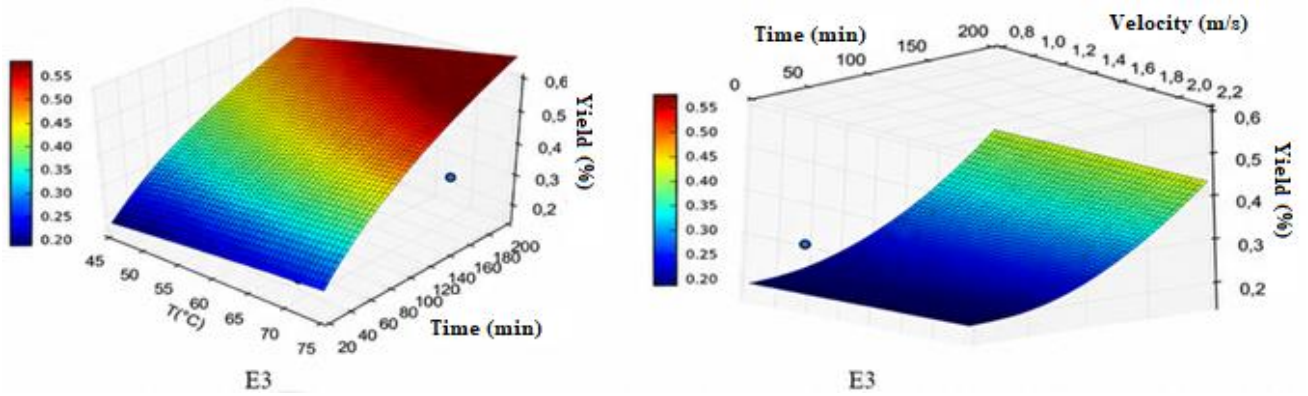


Figure 17. Response surface analysis of essential oil yield from orange peels (E1), obtained using Curv-Expert Professional.

Essential oil yield from all citrus varieties increased with higher temperature and longer drying time, with air velocity positively influencing yield under laminar flow conditions. Variety-specific responses were observed: E1 and E3 showed strong dependence on temperature and time, while E2 exhibited a more complex response with greater sensitivity to parameter interactions. Optimal yields were consistently obtained after 180 min, corresponding to the end of the falling-rate drying phase. Temperature was the most influential factor, followed by drying time, with air velocity having a secondary but positive effect. Overall, the maximum essential oil yield was achieved at 70 °C, 180 min, and 2 m/s air velocity.

3.5. Composition of Essential Oil of Citrus

Citrus essential oils primarily consist of volatile terpenes (Table 6). Major constituents include monoterpenes (D-limonene, α- and β-pinene, myrcene, sabinene), sesquiterpenes (β-caryophyllene, α-humulene), aldehydes (citral, citronellal), alcohols (linalool, α-terpineol, geraniol), and esters (linalyl acetate, geranyl acetate).

The chemical composition of the essential oils extracted from the peels of the three citrus species revealed that limonene was the predominant compound in all samples.

In *C. sinensis* peel oil, limonene accounted for 93% of the total composition, followed by α-pinene (2.4%), linalool (1.5%), and myrcene (1.3%). *C. limon* essential oil showed a lower limonene content (78%), with significant contributions from β-pinene (10.7%) and γ-terpinene (9.2%), in addition to minor amounts of sabinene (2%). In *C. aurantium*, limonene was overwhelmingly dominant (97%), while linalool (1.5%), β-pinene (1%), and myrcene (0.75%) constituted the remaining fraction.

These results highlight the species-specific variation in volatile profiles of citrus peels, with *C. limon* exhibiting a more diverse essential oil composition compared to the relatively monoterpene-rich profiles of *C. sinensis* and *C. aurantium*. The high limonene content observed, particularly in *C. sinensis* and *C. aurantium*, is consistent with previous studies and underscores the potential of these peel oils for applications in the flavor, fragrance, and pharmaceutical industries [22, 23].

Table 6. Major Chemical and Antioxidant Components of Citrus Essential Oils.

Species	Main Components	Composition (%)
Citrus sinensis		
	Limonene	93 ± 0.01
	Myrcene	1.3 ± 0.02
	α-Pinene	2.4 ±0.03
	Linalool	1.5 ±0.01
Citrus limon		
	Limonene	78 ± 0.04
	γ-Terpinene	9.2 ± 0.03
	β-Pinene	10.7 ±0.04
	Sabinene	2 ± 0.02

Species	Main Components	Composition (%)
Citrus aurantium		
	Limonene	97 ± 0.02
	Linalool	1.5 ± 0.02
	β-Pinene	1 ± 0.01
	Myrcene	0.75 ±0.01

3.6. Antimicrobial Activity After Thermal Drying

Fresh essential oils, particularly from *C. limon*, exhibited the strongest antimicrobial activity. Drying significantly reduced antimicrobial efficacy, likely due to volatilization or degradation of active compounds. Similarly, antioxidant activity decreased after drying, with air-drying showing the best preservation among the tested methods.

The disproportionate loss of antioxidant activity observed after thermal drying can be explained by the degradation of highly reactive minor terpenes, whose contribution to radical scavenging activity exceeds that of major hydrocarbons such as limonene.

Fresh E2 essential oil exhibited the broadest and most potent antimicrobial effect (Figure 18). It showed strong inhibition against *Candida albicans* (28 mm), *Klebsiella pneumoniae* (18 mm), *Salmonella arizonae* (18 mm), *Enterococcus faecalis* (14 mm), *Escherichia coli* (12 mm), and *Pseudomonas aeruginosa* (14 mm).

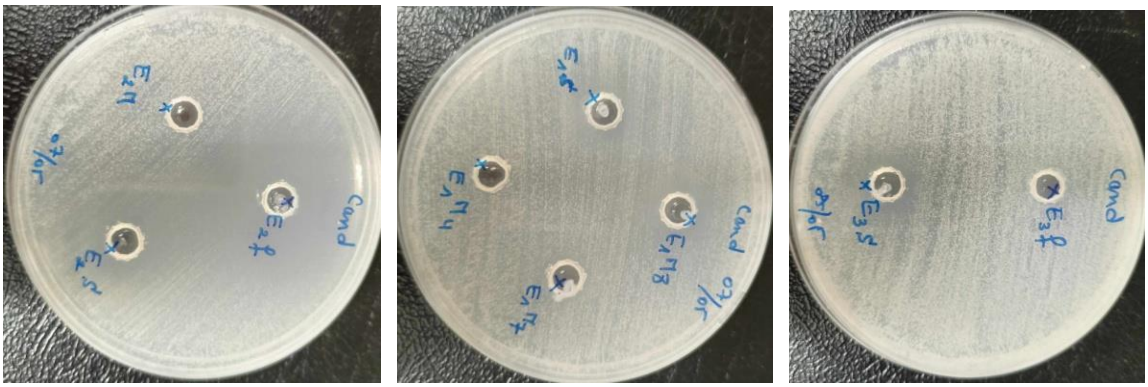


Figure 18. *Inhibition zones Candida albicans.*

Moderate inhibition was observed with air-dried E2 (Sall) against *Staphylococcus aureus* (14 mm) and with E1 M8 against both *C. albicans* (12 mm) and *S. aureus* (11 mm). All other dried samples, including those obtained by convective drying, showed little to no activity.

No antimicrobial activity was detected against *Listeria monocytogenes* or methicillin-resistant *Staphylococcus aureus* (MRSA) in any of the tested samples.

Essential oils from fresh citrus peels (particularly E2) demonstrated strong and broad-spectrum antimicrobial activity, whereas drying (air or convective) markedly reduced their efficacy. These findings highlight the importance of processing conditions, suggesting that fresh samples are preferable for maximizing antimicrobial potential.

3.7. Antioxidant Activities After Thermal Drying

Figure 19 illustrates the variation in IC_{50} values for the three citrus species in the fresh state and under different drying methods. Lower IC_{50} values correspond to higher antioxidant capacity.

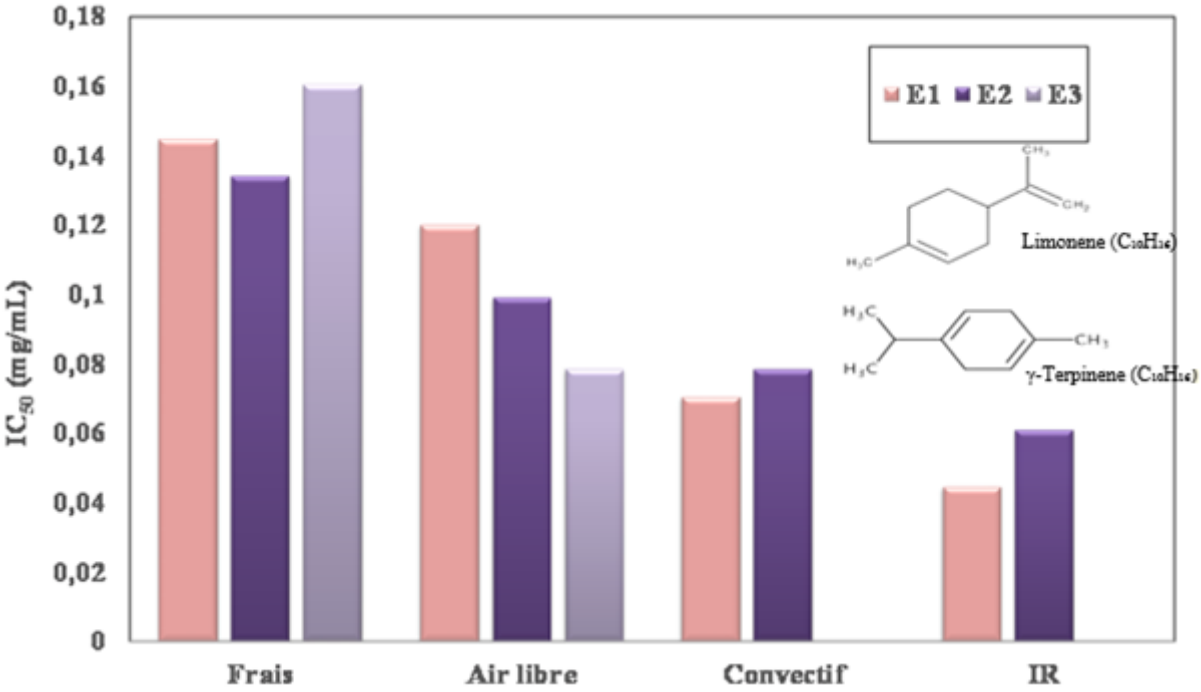


Figure 19. *Variation in IC_{50} values for the three citrus species in fresh state and under different drying methods.*

This figure shows a significant variation of the IC_{50} values for the three citrus species in fresh state and under different drying methods. In fact, the drying process significantly influences both the physicochemical properties and the chemical composition of citrus peels. Moisture removal during drying concentrates soluble solids, minerals, and bioactive compounds, but it can also induce the degradation or volatilization of thermolabile compounds. In particular, essential oils are highly sensitive to temperature and drying methods. Studies have shown that limonene, the dominant monoterpene in citrus peels, can decrease in content when peels are subjected to prolonged or high-temperature drying, while minor compounds such as linalool, myrcene, and β-pinene may be partially lost or transformed [22, 24, 25]. Similarly, antioxidant activity can be affected by drying: mild drying methods (e.g., freeze-drying or air-drying at low temperatures) tend to preserve phenolic and flavonoid content, whereas high-temperature drying can lead to a significant reduction in radical scavenging capacity [23, 26]. These observations underscore the importance of selecting appropriate drying conditions to maximize the retention of bioactive compounds and the functional properties of citrus peel by-products, particularly when the objective is to valorize them for industrial applications in food, pharmaceuticals, or cosmetics.

4. Discussion

From a valorization perspective, optimizing pretreatment conditions represents a key strategy not only to preserve the functional properties of citrus peel essential oils, but also to ensure batch-to-batch consistency, which is a critical requirement for industrial-scale applications in the food, cosmetic, and pharmaceutical sectors [3, 4].

The observed decrease in antioxidant activity following drying treatments is consistent with previous reports demonstrating the thermal degradation of phenolics and heat-sensitive terpenes [3, 12, 13]. Infrared drying, in particular, accelerates moisture removal but generates rapid surface heating, which may induce oxidative stress and loss of minor but bioactive constituents such as α-terpinene and γ-terpinene [18]. These minor terpenes, althoug6 present in low concentrations, are known to contribute disproportionately to radical scavenging capacity and antimicrobial activity [4, 11, 25].

Drying also modifies the volatile profile of citrus peel oils, altering the relative abundance of monoterpenes and oxygenated compounds [9, 16]. Our findings confirm that drying reduces both antimicrobial and antioxidant activities, emphasizing the importance of optimized pretreatment conditions to preserve functional properties. Mild convective drying, for instance, can limit oxidative degradation while facilitating oil release by weakening peel cell walls [6, 10].

Although limonene remains the predominant constituent of citrus essential oils, several studies have emphasized the disproportionate contribution of minor terpenes, such as α-terpinene and γ-terpinene, to antioxidant performance. The reduction of these highly reactive compounds during thermal drying may therefore explain the increased IC_{50} values observed in dried samples.

From an industrial perspective, these results underline the need for process-specific optimization. Combining moderate temperature, adequate drying time, and controlled airflow can

maximize oil yield while retaining bioactive compounds, enhancing the economic and functional value of citrus by-products [16]. This approach aligns with recent sustainable valorization strategies, highlighting the feasibility of producing high-quality essential oils at an industrial scale.

Finally, integrating drying optimization with downstream extraction and fractionation protocols could further enhance bioactive recovery, supporting applications in food preservation, cosmetics, and nutraceuticals [1, 5, 10].

5. Conclusion

This study demonstrates that citrus peel waste from Tunisian agriculture can be efficiently valorized through optimized drying and essential oil extraction processes. Species-dependent structural and drying behaviors were observed, highlighting the need for tailored processing conditions. Although fresh peels yielded essential oils with superior biological activities, optimized convective drying (70 °C, 180 min, 2 m s^{-1}) maximized oil yield while maintaining acceptable quality. These findings provide a scientific basis for the sustainable and industrial-scale valorization of citrus by-products in food, cosmetic, and nutraceutical applications.

Abbreviations

DoE	Design of Experiments
E1	Citrus Sinensis
E2	Citrus Limon
E3	Citrus Aurantium
Eos	Essential Oils
GC-MS	Gas Chromatography Coupled with Mass Spectrometry
IR	Infra Red
k	Number of Factors Studied
LB	Luria Broth
Mi	Initial Mass (g)
Mf	Final Mass (g)
M	Mass (g)
RV	Volumetric Shrinkage
RH	Relative Humidity (%)
T_c	Ash Content (%)
V	Volume (m^3)
WB	Winge Broth
X	Moisture Content ($g.g^{-1}$ Dry Matter)
X_i	Coded Values of the Independent Variables (Factors)
Y	Predicted Response (Essential Oil Yield (%))
β_0	The Intercept
β_i	The Regression Coefficients Representing the Effect of Each Factor
ε	Porosity (%)
ρ	Bulk Density

Author Contributions

Touil Amira: Conceptualization, Methodology, Project administration, Resources, Software, Validation, Writing – original draft, Writing – review & editing

HajAmmar Ahlem: Data curation, Investigation, Methodology, Resources, Supervision, Writing – original draft

Litaiem Jihene: Formal Analysis, Investigation, Resources, Visualization, Writing – original draft

Conflicts of Interest

The authors declare no conflicts of interest.

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