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## Transforming lemon Peel into a sustainable reservoir of bioactives: A green osmotic dehydration strategy

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#### ABSTRACT

Osmotic dehydration (OD) is a sustainable alternative, offering reduced energy consumption compared to traditional drying approaches. This study investigates the role of OD in stabilizing bioactive compounds in lemon peel, fostering sustainable citrus by-product applications. Employing Response Surface Methodology (RSM) framework, pivotal variables—temperature (30–60 °C), exposure time (60–180 min), and sucrose concentration (50–70 °Brix)—were optimized to enhance water loss (WL) and solid gain (SG) while safeguarding bioactive retention. The optimal conditions (58.92 °C, 70 °Brix, 159 min) yielded a WL of 3.4 g/g, SG of 1.5 g/g, and high sensory acceptability. The OD treated lemon peel powder exhibited substantial retention of bioactive compounds, including ascorbic acid (4.1 mg/g) and total phenols (2.3 mg gallic acid/g), surpassing untreated controls. This enhanced bioactive profile underscores its potential as a sustainable and functional ingredient in nutraceutical applications.

### 1. Introduction

The citrus industry worldwide generates approximately 120 million tons of agro-industrial residue annually, with peel, pulp and seeds constituting 50–60 % of the total fruit mass (Russo et al., 2021). These by-products are abundant in bioactive compounds, including essential oils, pectin, dietary fiber, and limonene, which possess significant potential for nutraceutical applications. *Citrus limon* is widely utilized in food, pharmaceutical, and cosmetic industries due to its potent antimicrobial, antioxidant, and antiviral properties. The processing of *C. limon*, however, generates lemon peel as a by-product, which is a rich source of bioactive compounds (Zema et al., 2018). It represents an exceptional reservoir of bioactive phytochemicals, comprising a diverse spectrum of

phenolic acids ( $5.9 \pm 0.4$  mg/g), including ferulic acid, p-coumaric acid, sinapic acid, caffeic acid, and chlorogenic acid, which are well-documented for their robust antioxidant, anti-inflammatory, and chemopreventive properties. Moreover, it exhibits a high concentration of flavonoids ( $18 \pm 2$  mg/g), such as naringin, hesperidin, and quercetin, known for their antioxidative efficacy, antimicrobial activity, and cardioprotective potential. Additionally, it is enriched with terpenoids, including p-limonene,  $\gamma$ -terpinene, and  $\alpha$ -citral, which impart characteristic organoleptic properties and exhibit significant anti-inflammatory, antifungal, and anticancer bioactivities. These bioactive constituents collectively highlight its utility as a promising natural resource for nutraceutical development and functional food fortification (Gómez-Mejía et al., 2019; Scurria et al., 2020). The recovery of

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bioactive compounds from peel, is encumbered by its complex matrix, which entraps valuable metabolites, thereby impeding efficient extraction. Furthermore, the inherent volatility of essential oils, which are susceptible to oxidative degradation, exacerbates extraction difficulties. Conventional extraction techniques often co-extract undesired contaminants, necessitating time-consuming purification. Additionally, the relatively low yields and the increasing demand for environmentally sustainable, green extraction methodologies further complicate the process, posing significant challenges for large-scale industrial applications. (Maqbool et al., 2023). Unlike traditional extraction techniques, Hydrodynamic cavitation offers a greener alternative with enhanced mass transfer, reduced solvent use, and preservation of thermolabile compounds. However, its adoption requires significant investment, precise optimization, and feasibility assessments based on substrate properties, with challenges such as high costs and water management limiting its broader application (Nuzzo et al., 2020).

Using the whole lemon peel, rather than isolated extraction, presents a viable alternative that minimizes compound degradation and avoids the use of toxic chemicals, aligning with green extraction principles (Jha & Sit, 2022). Limited studies focus on direct applications of whole lemon peel in food systems, yet such approaches can offer substantial benefits. For example, Moosavy et al. (2017) demonstrated that adding dried lemon peel powder to meat patties improved their sensory and physicochemical qualities. This evidence underscores the potential of lemon peel not only for its health-promoting effects but also in enhancing the functionality and nutritional value of food products.

Osmotic dehydration (OD) is widely acclaimed for its numerous advantages over traditional drying methods, such as minimizing heatinduced flavor and color loss, preserving bioactive compounds, reducing enzymatic browning, and decreasing energy costs. Pre-treating foods in an osmotic solution minimizes cellular damage, retains volatile flavors and acidity, and prevents structural collapse during drying, ultimately enhancing product quality (Ramya & Jain, 2017). Several citrus by-products, including orange peels, mandarin peels, kinnow peels, and kaffir lime peels have been extensively evaluated for preservation through osmotic dehydration (Kaur & Sharma, 2022; Lertworasirikul & Saetan, 2010; Mokrani et al., 2016; Sidhu et al., 2016). Sidhu et al. (2016) conducted a comprehensive assessment of the biochemical, microbial stability, and organoleptic properties of kinnow peel subjected to osmotic dehydration, in the form of candy and powder. Their findings indicated that osmotic dehydration effectively preserved the peel's pigmentation, aromatic compounds, flavor profile, and nutritional constituents, thereby facilitating prolonged storage and enhancing its shelf-life for consumption. Similarly, osmotic dehydration of orange peels has been proven to reduce moisture content while simultaneously preserving key bioactive compounds, such as antioxidants, thereby maintaining the peel's nutritional value and functional properties (Mokrani et al. (2016). These investigations underscore the potential of osmotic dehydration as a sustainable and effective method for enhancing the shelf life and maintaining the quality of food by-products, aligning with the broader goals of sustainable food processing and waste reduction.

Numerous studies have examined the effects of osmotic dehydration on whole lemon fruits (Lazou et al., 2020; Özkan-Karabacak et al., 2022; Sarkar et al., 2020). Despite its extensive application in various contexts, limited studies have explored the potential of osmotic dehydration for lemon peel. This study sought to systematically optimize the osmotic dehydration process for lemon peel utilizing response surface methodology (RSM), a sophisticated statistical approach for process enhancement. The central aim was to rigorously assess and improve the efficiency of the process by strategically modulating key variables, including processing duration, temperature, and sucrose concentration, while ensuring the retention of the bioactive compounds inherent to lemon peel. Furthermore, the physicochemical properties and nutritional attributes of the optimized OD treated lemon peel were meticulously compared with those of conventionally dried lemon peel,

enabling a comprehensive assessment of its potential to retain bioactive compounds and enhance functional value.

### 2. Methodology

### 2.1. Material

Fresh lemons (*Citrus limon*) and sucrose sugar were obtained from the local market in Tilak Nagar, situated in New Delhi, India.

### 2.2. Reagents and solvents

All chemicals and solvents used were of analytical grade, and distilled water was utilized throughout the study. Chemicals including 6-dichloroindophenol, sodium hydroxide, ascorbic acid, sodium bicarbonate, Folin-Ciocalteu reagent, and oxalic acid were sourced from Thermofisher, Mumbai, India.

### 2.3. Preparation of raw material

Sound ripe lemons on homogeneity in size and shape were selected. The lemons underwent a meticulous washing regimen with distilled water, repeated three times, followed by meticulous separation of the pulp and seeds from the peels. The isolated peels were subjected to thermal blanching in boiling water for 3 min to achieve enzymatic inactivation, color stabilization, microbial load reduction, and improved product efficiency, followed by immediate cooling in chilled water to arrest thermal effects (Kaur & Sharma, 2022).

### 2.4. Osmotic dehydration of lemon peels

The experiment was conducted using a central composite design within the framework of response surface methodology, comprising 17 distinct runs for Osmotic Dehydration (OD). The OD treatment was performed in a beaker containing osmotic solutions. Sucrose sugar syrup of the desired concentration was meticulously prepared, and its  $^\circ\textsc{Brix}$ value was measured using a Hand Refractometer. For each specific set of conditions, 50 g of lemon peels, mixed in a 1:4 ratio with sugar, were precisely weighed and introduced into the beaker for dehydration over a specified duration. The samples were subsequently extracted from the sugar solution and uniformly distributed onto tray dryers (fabricated tray dryer) where they underwent a dehydration process for 10 h at a controlled temperature of 60  $\pm$  5  $^{\circ}\text{C}$  to ensure optimal moisture reduction. Concurrently, control samples comprising untreated lemon peels were subjected to an identical drying protocol within the same tray dryer apparatus. Following this, the dried samples (OD treated and control) were finely minced using a kitchen grinder. The resulting powders were sieved through a sieve of 100 mm, packed into polyethylene bags, and stored at 20 °C until required for subsequent analysis. Subsequently, 5 g samples were drawn for the determination of three key responses: water loss (%), solid gain (%), and overall acceptance. The overall acceptance of the product has been assessed based on three sensory attributes: aroma, color, and appearance, to evaluate product acceptance. Water loss and solid gain were quantified using a drying oven, employing the equations outlined by Lertworasirikul and Saetan (2010).

$$\text{%Watert loss} = \frac{(MO - m0) - (Mt - mt)}{Mo}$$
 (1)

$$\%Solid Gain = \frac{mt - mo}{Mo}$$
 (2)

Where mo is the initial weight of solids (dry matter) of the sample (g), mt is the weight of solids (dry matter) of the sample after osmotic dehydration, Mo is the initial weight of the sample, and Mt. weight of sample after osmotic dehydration for time.

### 2.5. Experimental design

The statistical approach known as response surface methodology (RSM), based on analysis of variance (ANOVA), was applied to systematically evaluate the influence of three independent variables—total soluble solids (TSS) of OD solution, time, and temperature—on the critical dependent variables (weight loss, solid gain, and overall acceptance) of the process, using Design Expert Software version 13.0 (Statease Inc., Minneapolis, MN). The central composite design (CCD) incorporating three independent variables (Table 1) facilitated the experimental design. A quadratic model was developed to assess the effects of the interactions among the independent variables on the observed responses, namely overall acceptance (Y1), solid gain (Y2), and weight loss (Y3), of OD lemon peels as delineated in Eq. 1

$$Yi = \alpha o + \sum \alpha i Xi + \sum \alpha i i Xi^2 + \sum \alpha i j XiXj$$
 (3)

where Xi and Xj denote three independent variables (including TSS of OD solution, time, and temperature), Yi represents the response function of the dependent variable, encompassing parameters such as weight loss, solid gain, and overall acceptance. The terms  $\alpha 0$ ,  $\alpha i$ ,  $\alpha ii$ , and  $\alpha ij$  correspond to the constant, linear, quadratic, and interaction coefficients, respectively. The regression analysis was carried out to examine the model's effectiveness based on the regression coefficient (R²) and adjusted R.² Given the intricate nature of food products, an R² value exceeding 0.80 was considered indicative of a favorable fit to the model. Moreover, the optimized conditions (58.92 °C, 70°Brix and 159 min) derived from the statistical analysis were validated and subsequently compared against the control samples to comprehensively assess the physicochemical properties of the OD-treated lemon peel.

### 2.6. Physical parameters

$$\% Titratable \ acidity = \frac{0.1 \times equivalent \ weight \ of \ acid\% \times Normality \ of \ Na0H \times titre \ value}{Weight \ of \ sample} \tag{5}$$

### 2.6.1. Thickness of Peel

The thickness of OD treated lemon peel and control lemon peel was measured using a screw gauge with a least count of 0.01 mm. Measurements were taken at five randomly selected locations on each peel, and the mean thickness value was subsequently reported.

### 2.6.2. Bulk density

The bulk density of OD treated lemon peel powder and control lemon peel powder was determined by gently introducing a known mass of the powder into an empty graduated cylinder. The volume occupied by the powder was recorded, and this volume was used to calculate the bulk density, expressed in grams per milliliter (g/ml).

Bulk denisty = 
$$\frac{\text{weight of powder}}{\text{bulk powder volume}}$$
 (4)

**Table 1**Coded and experimental values of independent variables used in the CCD model.

|         | •                        |         |         |                       |                        |
|---------|--------------------------|---------|---------|-----------------------|------------------------|
| Symbols | Independent<br>Variables | Minimum | Maximum | Coded Low $(-\alpha)$ | Coded High $(+\alpha)$ |
| X1      | TSS of OD                | 43.18   | 76.82   | -1 ↔                  | +1 ↔                   |
|         | solution                 |         |         | 50.00                 | 70.00                  |
| X2      | Temperature              | 19.77   | 70.23   | -1 ↔                  | $+1 \leftrightarrow$   |
|         |                          |         |         | 30.00                 | 60.00                  |
| Х3      | Time                     | 19.09   | 220.91  | -1 ↔                  | +1 ↔                   |
|         |                          |         |         | 60.00                 | 180.00                 |

### 2.7. Proximate composition analysis

The proximate analysis of the samples was conducted in accordance with established protocols. The water content was evaluated using the methodology outlined by the Association of Official Analytical Chemists (AOAC (Association of Official Analytical Chemists), 2000). Briefly, 5 g of each sample was dried in a hot air oven until an equilibrium weight was achieved. The dried samples were then used to determine ash content by incinerating them in a muffle furnace maintained at 550 °C for 5 h. Crude protein content, calculated as a percentage of total nitrogen multiplied by 6.25, was determined using the Kjeldahl method with 2 g of each sample. Crude fat content was obtained by exhaustive extraction of 2 g of each sample using a Soxhlet apparatus, with petroleum ether (boiling range of 40–60 °C) as the extracting solvent. The carbohydrate content was determined using the difference method, as described by the equation:4.

Percentage (%) carbohydrate = 100 - (% moisture + % ash + % protein + % fat + crude fiber). Equation:4.

### 2.8. Chemical parameters

### 2.8.1. pH

The hydrogen ion concentration OD treated lemon peel powder and control lemon peel powder was measured using a digital pH meter (LT-10, Labtronics, Panchkula, Haryana).

### 2.8.2. % acidity

The percentage of anhydrous citric acid was determined using the volumetric method as specified by the AOAC (1995) and calculated using the provided formula.

### 2.8.3. Water activity

The free water present in OD treated and control lemon peel powder was estimated by placing a small amount of the sample in a water activity meter. (4te-Aqualab, Maharashtra, India).

### 2.8.4. Ascorbic acid content

The estimation of ascorbic acid content in both lemon peel powder samples (control and OD treated) was conducted using the volumetric method with 2,6-dichloroindophenol, as described in AOAC Method 967.2. In this procedure, 5 ml of a standard ascorbic acid solution (100  $\mu g/ml)$  was mixed with 4 % oxalic acid and standardized using 2,6-dichlorophenol indophenol dye. The endpoint of the titration was indicated by the appearance and persistence of a pink coloration. The volume of dye consumed (V1 ml) was recorded, representing the amount of ascorbic acid present in the standard solution. Similarly, the lemon peel powder samples were titrated against the dye (V2 ml). The ascorbic acid content in the sample was calculated using the appropriate formula.

$$Ascorbic \ Acid = \frac{0.5 \times V2 \times 100}{v1 \times 10 \times W} \times 100 \tag{6}$$

Where:

V1 = Volume of dye consumed during titration of standard solution (ml).

V2 = Volume of dye consumed during titration of sample (ml).

W = Weight of the sample (g).

This formula allows for the determination of the ascorbic acid content in the lemon peel samples.

### 2.8.5. Total phenolic content

The total phenolic content of both lemon peel powder extracts (OD treated and control) was determined using the modified Folin-Ciocalteu reagent (FCR) method (Al-Qassabi et al., 2018). Initially, a 10 % w/v methanolic solution of both OD treated and control lemon peel powder was left to stand for 120 min. The solution was subsequently centrifuged at 3500 rpm for 10 min to obtain a clear supernatant. Following this, 0.5 ml and 1 ml aliquots of each sample extract were mixed with 2.5 ml of distilled water. An equal volume of each extract and FCR reagent was combined and allowed to incubate for 3 min. To each test tube, 2 ml of 20 % sodium carbonate solution was added, and the tubes were then placed in a boiling water bath for 60 s. After cooling, the absorbance of the reaction mixture was measured at 650 nm. The phenolic content, expressed as milligrams of gallic acid equivalents (GAE) per 100 g of sample, was calculated by referencing a standard curve constructed using varying concentrations of gallic acid (0–1000  $\mu$ g/ml).

### 2.8.6. Electrical conductivity

The electrical conductivity of control lemon peel powder and OD-treated lemon peel powder was assessed by dissolving the contents in water and mixing them with the stick homogenizer. The solutions were incubated at 45  $^{\circ}\mathrm{C}$  for 40 min to facilitate hydration, and their electrical conductivity was subsequently measured to assess ion concentration post-rehydration. The electrical conductivity of both samples and tap water was measured at room temperature using a portable EC/TDS conductometer.

### 2.8.7. Fourier transform infrared spectroscopy (FTIR)

The FTIR spectra were obtained for OD lemon peel powder and control lemon peel powder using FTIR spectroscopy (Bruker, Alpha E FTIR, Germany). It is a non-invasive, rapid, and efficient method utilized for identifying various structural moiety. The spectra were recorded as absorbance (%) across wavelengths ranging from 500 to 4000 cm<sup>-1</sup>.

### 2.9. Statistical analysis

The results are expressed as mean  $\pm$  standard deviation. Analysis of variance (ANOVA) was conducted to assess the significance of treatment effects, followed by Tukey's multiple comparison test. Statistical analyses were performed using SPSS software with a significance threshold set at P < 0.05 (Ping et al., 2024).

### 3. Results

### 3.1. Fitting of the response surface model

Second-order polynomial equations were derived in the experimental model, with 17 experimental runs conducted and performed in duplicate. The studied responses for the design included solid gain, weight loss, and overall acceptance. The adequacy of the fitted model for the studied response was assessed through the high value of the coefficient of determination  $(R^2)$ , as shown in Table 2.

### 3.2. Effect of independent variable on the osmotic dehydration of lemon peel

### 3.2.1. Overall acceptance

A statistical analysis of variance was executed to ascertain the significant impacts of process variables on the overall acceptance response. Specifically, the treatment time variable, both in linear and quadratic forms, demonstrated a statistically significant response on the overall acceptance (p < 0.05). Variables that did not attain significance (p > 0.05) were systematically removed from the models without disrupting the model structure. Furthermore, the comprehensive analysis conducted in this study indicated that the constructed models adequately captured the variability in the data and effectively represented the relationship between the variables of interest, as evidenced by the insignificant lack of fit.

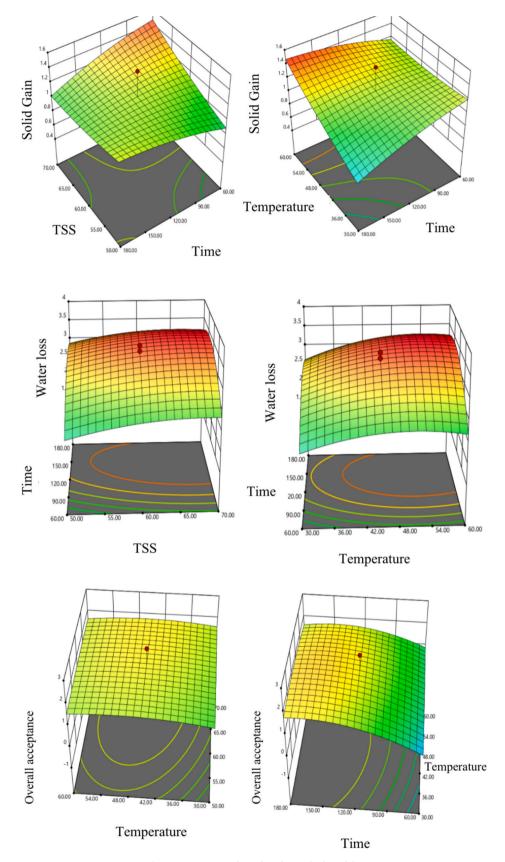
The holistic evaluation of osmotically dehydrated products, including all sensorial attributes, serves as a comprehensive metric. da Costa Ribeiro et al. (2016) underscored a similar perspective, emphasizing that osmotic dehydration conducted before convective drying contributes significantly to the preservation of quality and sensory characteristics in dehydrated pear. Notably, treatment time emerges as a pivotal determinant influencing the alignment of the product with consumer preferences and expectations. A decrease in immersion time negatively impacts the overall acceptability of osmotically dehydrated lemon peel. This phenomenon may be attributed to the increase in soluble solids with prolonged immersion time at 50–70°Brix, particularly in contrast to temperature variations Yadav et al., 2012. Furthermore, an increase in sucrose content, as suggested by Özbek, 2023, is posited to mitigate the bitter flavor of the peel, thereby enhancing consumer acceptance.

### 3.2.2. Solid gain

Surface graphs present in Fig. 1 illustrate the integrated impact of the two process variables on solid gain, while holding the other process

**Table 2**Central composite design with regression coefficients.

| Dependent Variables   | Solid Gain |        |         | Weight Loss |        | Overall Acceptance |           |        |         |
|-----------------------|------------|--------|---------|-------------|--------|--------------------|-----------|--------|---------|
|                       | df         | SOS    | p-value | COF         | SOS    | p-value            | COF       | SOS    | p-value |
| Model                 | 9          | 5.68   | 0.0448  | 3.26        | 7.85   | 5.68               | -5.1827   | 5.68   | 0.0448  |
| X1-TSS of OD solution | 1          | 0.1389 | 0.3882  | 0.1990      | 0.5410 | 0.1389             | 0.06794   | 0.1389 | 0.3882  |
| X2-Temperature        | 1          | 0.0744 | 0.5224  | 0.2452      | 0.8211 | 0.0744             | 0.10788   | 0.0744 | 0.5224  |
| X3-Time               | 1          | 3.39   | 0.0026  | 0.4620      | 2.91   | 3.39               | 0.02615   | 3.39   | 0.0026  |
| X1X2                  | 1          | 0.0153 | 0.7689  | 0.0187      | 0.0028 | 0.0153             | -0.0002   | 0.0153 | 0.7689  |
| X1X3                  | 1          | 0.1653 | 0.3489  | -0.0438     | 0.0153 | 0.1653             | 0.00024   | 0.1653 | 0.3489  |
| X2X3                  | 1          | 0.1431 | 0.3814  | 0.0537      | 0.0231 | 0.1431             | -0.0001   | 0.1431 | 0.3814  |
| $X1^2$                | 1          | 0.0423 | 0.6273  | -0.1405     | 0.2226 | 0.0423             | -0.0006   | 0.0423 | 0.6273  |
| $X2^2$                | 1          | 0.3222 | 0.2038  | -0.2483     | 0.6953 | 0.3222             | -0.0007   | 0.3222 | 0.2038  |
| $X3^2$                | 1          | 1.65   | 0.0156  | -0.5506     | 3.42   | 1.65               | -0.000106 | 1.65   | 0.0156  |
| Residual              | 7          | 1.15   |         |             | 0.4671 | 1.15               |           | 1.15   |         |
| Lack of Fit           | 5          | 0.4305 | 0.9140  |             | 0.3890 | 0.4305             |           | 0.4305 | 0.9140  |
| Pure Error            | 2          | 0.7181 |         |             | 0.0781 | 0.7181             |           | 0.7181 |         |
| Total                 |            | 1.47   |         |             | 8.32   |                    |           | 6.83   |         |
| $R^2$                 | 16         | 0.8355 |         |             | 0.9438 |                    |           | 0.8318 |         |
| Adj- R <sup>2</sup>   |            | 0.6241 |         |             | 0.8716 |                    |           | 0.6154 |         |
| CV                    |            | 15.80  |         |             | 10.31  |                    |           | 31.87  |         |



 $\textbf{Fig. 1.} \ \ \text{Response surface plots for studied models.}$ 

variable at central values. In the early stages of the process, the osmotic driving forces exert significant pressure between the concentrated solution and the fresh sample, thereby accelerating the rate of water removal and solid gain.

Notably, the temperature variable exhibits a direct relationship with the solid gain of lemon peel in the osmotic dehydration process. It has been documented that an elevation in temperature throughout the osmotic dehydration process markedly enhances the rate of solid accumulation within the product. As temperature increases, the kinetic energy of molecules accelerates, thereby augmenting the diffusion coefficients of solutes and water, which facilitates a more pronounced influx of solids into the cellular matrix. This thermal augmentation leads to a significant escalation in solid gain over time, as elevated temperatures intensify the osmotic gradient, promoting enhanced mass transfer and solute uptake, which in turn alters the physicochemical properties and structural integrity of the treated product (Kaur et al., 2022). Accordingly, the rate of solid gain increased to a certain extent by accelerating the immersion temperature and time, after which it reached equilibrium and then declined at a specific sucrose concentration (Sidhu et al., 2016). However, the augmentation in solid gain was significantly attenuated by temperature elevation compared to the effect of time, as the sucrose concentration ascended from 50 to 70°Brix. This observation aligns with a study conducted by Yadav et al. in (2012), focusing on the optimization of peach using a response surface methodology (RSM) approach.

### 3.2.3. Water loss

An analysis of variance was undertaken to assess the significant effects of process variables on water loss (WL). All response variables were found to be statistically important in the case of WL, excluding the interaction effect responses. During the drying process, a direct relationship was observed between temperature and dehydration efficiency, with increasing temperature resulting in higher dehydration efficiency. This led to achieving a moisture content of 12.35 g/100 g and a water activity of 0.63 after processing at 60  $^{\circ}\text{C}$  for 3 h.

Several researchers have reported the expedited evaporation of water in the initial stages of osmotic dehydration (Kaur & Sharma, 2022; Mokrani et al., 2016; Yadav et al., 2012). The maximum water loss was achieved at 60 °C immersed in sugar solution for 3 h. The rising temperature over processing time reinforces water loss. Notably, higher process temperatures facilitate rapid water loss, hereby diminishing the duration of immersion needed to attain equilibrium concentration (Mokrani et al., 2016). The product weight loss increases with an increase in temperature and sucrose concentration. The decrease in weight loss observed at high temperatures over an extended duration could be due to the influence of temperature on the cell membrane semipermeability. This effect leads to the plasticization and swelling of the cell membrane, consequently reducing the rate of osmosis (Sidhu et al., 2016). Additional factors, such as a decrease in the density of the sucrose solution and enhanced diffusion coefficient of water at higher temperatures, further contribute to the accelerated rate of water migration (Yadav et al., 2012).

### 3.3. Optimization of osmotic dehydration process for lemon peels

In food industry-based processes, optimization involves multiple conditions that encompass quality characteristics and performance measures of the systems. Achieving this optimization entails maximizing or minimizing the process variables through a desirability approach, aimed at producing high-quality products efficiently.

The optimum condition for osmotic dehydration of lemon peels was determined to obtain maximum water loss and overall acceptance and minimum solid gain as specified by Kaur et al., (2022). Second-order polynomial equations were formulated to analyze the impact of independent variables on the response and identify the optimal conditions.

In this study, temperature, processing time, and total soluble solids

were selected in the range 30-60 °C, 60-180 min, and 50-70° Brix respectively. After applying the desirability function method, the obtained optimum conditions for the solution were 70° Brix sucrose solution at 59 °C temperature for 153 min. The predicted property of OD lemon peel in optimized solution indicated weight loss of 3.4 g/g, 1.5 g/ g of solid gain, and 0.8 scores for overall acceptance. The overall desirability was found to be 0.915. The optimized processing parameters were meticulously validated to ensure precision and reliability. A comparative evaluation between the empirical data and the theoretically optimized values demonstrated no statistically significant deviations, corroborating the robustness and predictive accuracy of the optimization framework. This congruence underscores the model's efficacy in forecasting experimental outcomes with remarkable fidelity, thereby affirming its practical utility. The osmotically dehydrated lemon peel in optimized conditions was further evaluated for various physicochemical changes to study their effect on different properties.

### 3.4. Proximate composition analysis

The nutritional analysis of OD treated lemon peel powder is presented in Fig. 2. A statistical difference (p < 0.05) has been observed in the case of all the proximate (moisture, carbohydrate, protein, crude fat, ash content) except crude fiber. In comparison to conventionally dried lemon peel powder, the mean moisture content of OD-treated lemon peel powder was found to be higher. This observation aligns with the water activity data of OD-treated lemon peel presented in Table 3. The osmotic dehydration process effectively reduces moisture content; however, the presence of a high concentration of solutes, such as sugars, in the cells binds with water molecules, leading to their retention in the cells.

The total carbohydrate content of OD-treated lemon peel powder was significantly higher compared to control lemon peel powder. This increase can be attributed to the sucrose syrup absorbed by the peel cells during the osmotic dehydration process through osmosis. Similar findings were reported by Pahari et al. (2022), where an up to 80 % increase in carbohydrate content was observed in papaya candy.

Conversely, other proximate such as protein, crude fat, ash, and crude fiber exhibited a decrease of 16 %, 32.5 %, 19 %, and 9 %, respectively, compared to control lemon peel powder. This reduction may be attributed to the osmosis process, wherein the migration of compounds from the cells results in the loss of certain nutrients. Similar findings regarding a reduction in the concentration of nutrients during osmotic dehydration were also reported by Araya-Farias et al. (2014), particularly in the case of osmotically dehydrated seabuckthorn fruits. This reinforces the notion that the OD method can lead to alterations in the proximate composition of various fruits, depending upon the specific process variables and the type of product being treated. The variability in outcomes underscores the importance of considering and optimizing process parameters to achieve desired nutritional characteristics in osmotically dehydrated fruits.

All the parameters are on a dry basis except moisture.

### 3.5. Physical properties

### 3.5.1. Thickness of peel

The comparative analysis of lemon peel thickness between the control sample and lemon peel subjected to OD treatment is elucidated in Table 3. There is no significant difference in mean thickness between the control lemon peel and OD-treated lemon peel. This suggests that the method of dehydration, whether conventional or osmotic, does not result in a statistically significant difference in the thickness of the lemon peel. Remarkably, the OD-treated lemon peel exhibited a substantial increase in thickness following a 153-min immersion in a sucrose solution. The observed increase in peel thickness can be attributed to the inherent porous structure of lemon peel that facilitates the absorption of the sugar solution, thereby resulting in both swelling and a discernible

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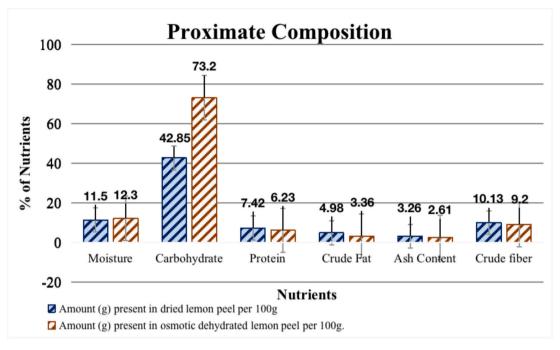


Fig. 2. Proximate composition of dried and Osmotically dried lemon peel powder.

**Table 3**Physical and chemical parameters of OD treated and control Lemon Peel.

| Physi     | Physical Parameters  |   |   |  |  |  |
|-----------|--|---|---|--|--|--|
| S.<br>No. | Parameters   | Control Lemon peel                          | OD-treated lemon peel                     |  |  |  |
| 1.        | Thickness (peel)   | $1.8 \pm 0.25 \text{ mm}$                   | $3.6 \pm 3.72 \text{ mm}$                 |  |  |  |
|           | 2. Bulk Density (powder) $0.471 \pm 0.34$ $0.752 \pm 0.81$ Chemical Parameters |   |   |  |  |  |
| 3.        | % Acidity  | $0.41~\% \pm 0.39$                          | $0.58~\%\pm0.64$                          |  |  |  |
| 4.        | pН   | $5.04 \pm 2.95$                             | $4.23 \pm 3.76$                           |  |  |  |
| 5.        | Ascorbic Acid  | $0.648 \pm 0.45\text{mg/g}$ of powder       | $4.1\pm3.01\mathrm{mg/g}$ of powder       |  |  |  |
| 6.        | Total phenolic content (Gallic acid)   | $0.828 \pm 0.01$ mg gallic acid/g of powder | $2.3 \pm 0.91$ mg gallic acid/g of powder |  |  |  |
| 7.        | Water Activity   | $0.48\pm0.32$ at $32.07^{\circ}\text{C}$    | $0.63 \pm 0.81$ at $30.79$ °C             |  |  |  |

Values are the means  $\pm$  S.D. of triplicate determination.

increase in peel thickness.

### 3.5.2. Bulk density

Bulk density characterizes the behavior of a substance in its dry solid state and its volume occupancy during the packaging process. The determination of bulk density for powdered products is of paramount importance, as it yields valuable insights into the quantity of product that can be accommodated within a specific volume of packaging material. The particle size, structures, and moisture content of powdered products are a few factors that substantially influence bulk density (Tekgül & Baysal, 2018). It is noteworthy that a negative correlation exists between particle size and the bulk density of dehydrated powders, where smaller particles enhance the cohesiveness of the desiccated sample, resulting in a more densely packed product (Farahmandfar et al., in 2020).

In this study, the bulk density of the control lemon peel powders was determined to be 0.47 g/ml, but this value significantly increased to 0.752 g/ml following exposure to OD treatment. The analysis of bulk density revealed a statistically significant difference between the control lemon peel powder and the OD treated lemon peel powder, underscoring the impact of the dehydration process on this critical property. The

observed increase in bulk density in OD-treated samples can be attributed to the heat treatment applied during osmotic dehydration, which was absent in the control samples (Farahmandfar et al., 2020). Elevated bulk density enhances packaging efficiency by accommodating a greater product volume per unit space, thereby reducing the consumption of packaging materials and associated transportation costs. Additionally, it aligns with sustainable practices by reducing the environmental impact associated with packaging and logistics (Versino et al., 2023).

### 3.6. Chemical parameters

### 3.6.1. pH

The pH level is a critical parameter indicative of the acid concentration within a food sample. In this study, it has been observed that the pH value of OD-treated lemon peel powder was marginally lower compared to control lemon peel powder (Table 3). Notably, lemon peels subjected to drying without OD treatment experience a substantial loss of acids during the dehydration process when compared to fresh lemon peels with a pH of 4.5  $\pm$  0.01 (Pham et al., 2020). This observed difference in pH levels could be attributed to the protective influence of sugars on acids or the diminished extent of heat exposure in OD-treated samples relative to conventionally heated counterparts. These findings resonate with those documented by Zhao et al. (2014) in their investigation on freeze-dried OD-treated mango, wherein they underscored the preservation of inherent quality attributes and nutritional content. Furthermore, Peiró et al. (2006) have postulated that the decline in pH may contribute to inhibiting the proliferation of microorganisms, while the presence of acids within the solution could also positively influence the mitigation of enzymatic browning processes.

### 3.6.2. Titratable acidity

The citric acid content in osmotically treated lemon peel powder, measured using a volumetric method, ranged from 0.41 % to 0.58 %. Notably, the acidity values of OD-treated samples were found to be significantly high compared to control dried samples. This trend aligns with similar observations reported by Toor and Savage (2006) in the case of OD-treated tomatoes and by Peiró et al. (2006) in the case of OD-treated grapefruits. However, it is noteworthy that a reduction in total acidity was detected in OD treated lemon peel powder in comparison to

fresh lemon peel (0.63 %) (Pham et al., 2020). This phenomenon may be attributed to the leaching of acids into the medium during the osmotic dehydration process, as suggested by Phisut et al. (2013).

### 3.6.3. Water activity

Water activity serves as a critical parameter profoundly impacting the preservation duration of dehydrated powder products. Food items with a free water content of less than 0.6 water activity (aw) are generally regarded as being in a state of microbiological stability (Tekgül & Baysal, 2018). This study identified a statistically significant variation in the water activity levels between the OD treated lemon peel powder and the untreated control lemon peel powder. The water activity of the control lemon peel powder measured was 0.48, aligning closely with the outcomes proposed by Tekgül and Baysal (2018) for dehydrated lemon peel powder. However, the osmotic dehydration process produced a substantial and notable increase of 31.25 % in the water activity of lemon peel powder. This trend mirrors the findings of Lazou et al. (2020), wherein their research revealed a parallel effect in the case of OD-dried tomatoes.

### 3.6.4. Total phenolic content

The total phenolic content (TPC) serves as a critical indicator of antioxidant capacity with significant implications for both human health and plant physiology. OD-treated lemon peel powder exhibited a statistically significant (p < 0.05) elevation in TPC content, with a notable increase of approximately 64 % compared to control lemon peel powder. However, OD-treated samples demonstrated a decrease in TPC content relative to fresh lemon peel (0.64 mg/g) (Gómez-Mejía et al., 2023). This decline in TPC during drying processes is attributed to the susceptibility of polyphenols to heat-induced degradation and the potential formation of irreversible chemical modifications. Additionally, interactions between polyphenols and other compounds, such as proteins, may hinder their extraction or determination using conventional analytical methods. Leaching of polyphenols into the osmotic solution during processing may further contribute to their reduction, especially under conditions that favor mass transfer. Furthermore, the inactivation of oxidative enzymes, such as polyphenol oxidase and peroxidase, during the drying process can exacerbate the degradation of polyphenols and other bioactive compounds present in the samples, a phenomenon corroborated by findings from Özkan-Karabacak et al. (2022).

### 3.6.5. Ascorbic acid

A statistically significant (p < 0.05) increase in ascorbic acid content was observed in OD-treated lemon peel powder compared to control lemon peel powder. This increase was consistent with other parameters examined in the study, such as pH and % titratable acidity. Nevertheless, a significant reduction of 29 % in ascorbic acid content was observed in OD treated samples compared to fresh lemon peel (Vilela et al., 2016). This diminution in ascorbic acid levels can be ascribed to its inherent thermal instability, as the application of elevated temperatures during processing induces its degradation. (Özkan-Karabacak et al., 2022). Food samples subjected to a hypertonic osmotic solution and lower drying temperatures exhibited increased levels of ascorbic acid. This observation suggests that hypertonic osmotic solutions may inadequately encapsulate the sample, resulting in increased leaching of ascorbic acid into the medium during the drying process (Sarka et al., 2020) Nonetheless, Naga et al. (2015) suggested that the addition of 1 %ascorbic acid to the osmotic agent, such as sucrose, may aid in retaining ascorbic acid levels in the food sample.

### 3.6.6. Electrical conductivity

Electrical conductivity serves as a non-destructive method for assessing the quality characteristics of food samples, as discussed by Prisacaru et al. (2023). In the realm of effectively simulating electrical heating processes, such as ohmic and microwave heating, it is generally imperative to conduct electrical tomography studies (Subbiah &

Morison, 2018). This measurement reflects the concentration of soluble solids and ions in the medium, signifying the dissemination of intercellular ions from the cells. This release can be attributed to the catalysis of cellular damage or the formation of a porous structure within the material during the drying process.

The electrical conductivity of food samples is contingent upon their chemical composition, which encompasses factors like ionic concentration, the presence of oils, mineral content, and sugar levels. In the context of this study, it was observed that the control lemon peel powder exhibits higher electrical conductivity in comparison to the OD-treated lemon peel powder (Table 4). The variation is likely due to the elevated TSS and sugar content in the OD-treated lemon peel powder. Increased solute concentration augment the viscosity of osmotic solution, hindering ion mobility within the matrix. As TSS concentration intensifies, the solution becomes more saturated, restricting available volume for ion diffusion. Additionally, the higher sucrose content, interacts with water molecules, further impeding ion diffusion and lowering electrical conductivity. This reflects the intricate interplay between solute concentration, viscosity, and mass transfer dynamics (Castro et al., 2003) However, a discrepancy in the result of conductivity has arisen in the case of pomegranates where pomegranate juice was used as OD agent. This was perhaps due to the solids present in pomegranate juice retained during the OD treatment were not permanently attached to the aril matrix, meaning they could potentially be separated from it, affecting the observed results (Cano-Lamadrid et al., 2017).

### 3.6.7. FTIR analysis

Fourier Transform Infrared (FTIR) spectroscopy was employed to analyze the chemical bonds present in both control lemon peel and OD treated lemon peel Fig. 3. Examination of the spectra depicted in Fig. 3 reveals an increased sucrose concentration in the OD-treated lemon peel, as indicated by the pronounced peak at 995 cm<sup>-1</sup> (Leopold et al., 2011). Additional peaks within the 900–1153 cm<sup>-1</sup> range correspond to the C—O and C—C stretching vibrations, whereas peaks in the 1400–1199 cm<sup>-1</sup> range are attributed to the bending vibrational modes of O–C–H, C–C–H, and C–O–H in carbohydrates.

Furthermore, the occurrence of peaks at approximately 2900 cm<sup>-1</sup>, 1700 cm<sup>-1</sup>, and 1100 cm<sup>-1</sup> in the OD-treated lemon peel powder spectra may encompass spectral characteristics attributed to the presence of C—H, C=O, and C—O structures. These peaks suggest the presence of terpenoid constituents, such as limonene and ascorbic acid, respectively (Benoudjit et al., 2020). Moreover, additional stretching observed at 3592.31 cm<sup>-1</sup> in OD-treated lemon peel powder indicates a high pectin content (Kanmani et al., 2014).

### 4. Conclusion

Osmotic dehydration represents a cutting-edge preservation methodology characterized by reduced thermal treatment, thereby mitigating energy consumption while introduces novel flavor profiles and sensory attributes to the food matrix. *Citrus limon*, a widely utilized fruit, generates significant waste in the form of peel, pomace, and seeds. The peel, often discarded as a by-product of the juice industry, is, however, rich in valuable functional compounds and dietary fiber. This study demonstrates that optimizing the osmotic dehydration process for lemon peel results in elevated antioxidant levels, with notably higher concentrations of ascorbic acid and phenolic compound\, owing to the milder temperature conditions employed. In contrast, conventional drying

**Table 4**Conductivity of OD treated lemon peel powder.

| S.No. | Sample                       | Resistivity | Conductivity  |
|-------|------------------------------|-------------|---|
| 1.    | Distilled Water              | 190-200 k Ω | $\begin{array}{c} 5.12 \times 10^{-3} k\Omega \\ 2.2 \times 10^{-2} \; k\Omega \\ 5.4 \times 10^{-3} k\Omega \end{array}$ |
| 2.    | Dried Lemon Peel powder      | 44-45 k Ω   |   |
| 3.    | OD treated Lemon peel powder | 180–190 kΩ  |   |

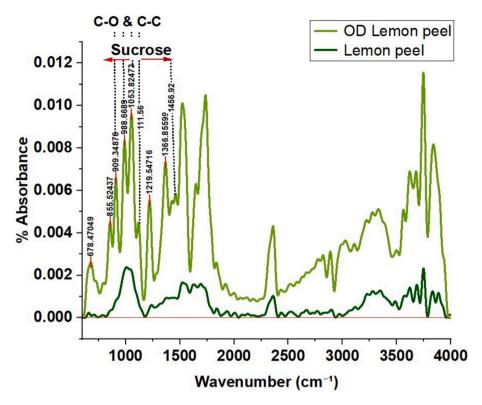


Fig. 3. FTIR analysis of OD treated and control lemon peel powder.

methods applied to lemon peel lead to the degradation of these functional compounds. Furthermore the resultant OD treated lemon peel powder exhibits a candied texture, rendering it suitable for direct consumption or incorporation into digestive spice blends and nutraceutical products. However, further investigative research is necessary to evaluate the potential presence of anti-nutritional factors and the impact of lemon peel consumption on gut microbiota composition. Investigating the scalability of the optimized process for industrial applications and assessing its economic feasibility in comparison to traditional drying methods could significantly advance its commercial viability. Moreover, exploring the synergistic effects of OD-treated lemon peel with other functional ingredients in the formulation of fortified foods or supplements may unlock new avenues in the development of sustainable and health-focused food products.

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### CRediT authorship contribution statement

Priyanka Prajapati: Writing – original draft, Visualization, Validation, Methodology, Investigation, Formal analysis, Conceptualization. Chhavi Porwal: Writing – original draft, Methodology, Investigation. Meenakshi Garg: Writing – review & editing, Visualization, Conceptualization. Neha Singh: Writing – review & editing, Investigation. Susmita Dey Sadhu: Writing – review & editing, Investigation. Rajni Chopra: Writing – review & editing. Eram S. Rao: Writing – review & editing, Validation, Investigation. Aparna Agarwal: Writing – review & editing, Conceptualization. Mohd Saeed: Writing – review & editing. Safia Obaidur Rab: Writing – review & editing. Dipendra Kumar Mahato: Writing – review & editing. Pradeep Kumar: Writing – review & editing. Madhu Kamle: Writing – review & editing. Abhishek Dutt

Tripathi: Writing - review & editing.

### **Declaration of competing interest**

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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### Data availability

No data was used for the research described in the article.

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