

High-value calcium chloride production from chicken, duck, and ostrich eggshell waste: A comparative analysis of different eggshells

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ABSTRACT

This research explores extracting calcium chloride from eggshell waste, a sustainable food additive that helps combat calcium deficiency. It emphasizes the potential of utilizing eggshells—often discarded as waste—due to their prevalence in diets worldwide. Chicken, duck, and ostrich eggshells were treated with 4% hydrochloric acid and dried using oven, spray, and freeze-drying methods. Thermogravimetric analysis (TGA) revealed that spray-dried chicken eggshells contained significant calcium carbonates, making them suitable for calcium chloride production. TGA and differential scanning calorimetry (DSC) analyses revealed mass loss, indicating significant dehydration at 260 °C, with thermal stability observed between 70 and 160 °C. Notably, the sample weight began to increase at temperatures around 100 °C, suggesting the presence of bound water in the crystal lattice. FTIR showed similar organic compositions among CaCl₂ powders, with variations in crystalline structures based on eggshell sources. SEM confirmed that the powders exhibited nearly spherical morphology. The yield of CaCl₂ was highest from chicken oven-dried powder, while spray-dried CaCl₂ exhibited higher flowability. Freeze-dried and spray-dried powders showed higher purity than oven-dried samples, as prolonged high temperatures in oven-drying affected mineral structures, reducing purity. The eggshell source did not significantly impact purity. Oven-dried powders had lower whiteness index and higher total color change (ΔE), influenced by particle size. All dried powders exhibited higher ΔE values than Control's, likely due to chemical reactions during heating. Overall, these methods enhance economic feasibility and align with circular economy principles, suggesting further research to optimize processes and evaluate CaCl₂ applications.

1. Introduction

Massive amounts of solid waste, including organic waste, are generated globally, leading to environmental issues (Gupta et al., 2022). Food processing wastes, particularly from the egg industry, contribute significantly to this waste stream. The production of various food products, including pastries and baked goods, results in substantial disposal costs due to the generation of eggshell waste (Chahal et al., 2021). Food processing wastes are the finished goods from different food processing companies that haven't been recycled or used for another purpose. They consist of raw material byproducts whose

economic value is lower than the costs associated with collecting and recovering them for reuse, leading to their disposal as waste (Duenas & Garcia-Estevez, 2020). These wastes include considerable amounts of bioactive elements, including phenolic compounds, dietary fibers, minerals, carotenoids, polysaccharides, vitamins, pigments, and lipids, which have been attributed to improved health (Mislata et al., 2020). Food waste could be seen as valuable by-products if proper management and technical methods were implemented, and if the value of the resulting products outweighed the costs of reprocessing. The fruit and vegetable, oil, seafood, and dairy industries are the main producers of a large quantity of food waste worldwide. This waste is generated at

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several phases of food processing, such as pre-harvest, post-harvest, manufacturing, packing, and at the consumer or home level (Ezzat et al., 2022).

Eggs from chickens are commonly used as ingredients in factory-processed foods. Currently, they are incorporated into a variety of products, including pastries, baked goods, and salad dressings. The production of these items leads to the generation of several tons of waste eggshells daily, resulting in substantial disposal costs worldwide. According to the FAO, approximately 8638,779 tons of eggshells are produced annually (Sivakumar et al., 2022). Eggshells contain a high concentration of calcium carbonate (CaCO_3), typically ranging from 92% to 95% by weight. This compound can serve as a firming agent in dairy production, aiding in coagulation, enhancing the cheese-making process, and increasing the firmness of cheeses. Additionally, it can be utilized as a preservative and curing agent in the preservation of fresh-cut fruits and vegetables (Strelec et al., 2024). An essential ingredient for many biological processes, including blood coagulation, chronic illnesses, muscle and nerve cell development, and mitosis, is calcium. The recommended daily consumption of calcium is 800–1300 mg, depending on the consumer's age. Milk and dairy products are typically thought of as the finest natural sources of calcium, with between 75% and 89% of the calcium coming from these sources. Given the nutritional content of dairy products, calcium derived from these sources has a high bioavailability and may be supplied at a comparatively cheaper cost. Researchers have shown that adding calcium to a variety of foods can be a cost-effective method of getting more calcium while also guaranteeing that the body gets all the calcium it needs (Domrongpakkaphan & Khemkhao, 2017).

Calcium chloride has been used extensively as a firming agent in various fruit and vegetable products and as a thickener in dairy products. Its application in calcium chloride solution injection and marinating improves the postmortem tenderization of beef steaks and enhances the meat's ability to retain water. Given the importance of calcium in the human diet, the food industry is continually exploring alternative calcium sources and developing new calcium-fortified products to meet consumers' daily calcium requirements. The production of calcium chloride from eggshell waste presents a sustainable option for utilizing a natural resource that is often disposed of as waste. This process not only adds value to the eggshells but also addresses the increasing demand for calcium in various applications. By converting eggshell waste into calcium chloride, we can contribute to more sustainable practices within the food industry and beyond, providing an alternative source of calcium that aligns with current health and environmental priorities (Garnjanagoonchorn & Changpuak, 2007; Jalali et al., 2024).

To date, there have been no reports on the extraction and production of calcium chloride from duck and ostrich eggshells using freeze and spray-drying methods. The goal of this research was to prepare a CaCl_2 solution from chicken, duck, and ostrich eggshells using hydrochloric acid (HCl). This solution was then dried using various methods (freeze-drying, spray-drying, and oven-drying) to enhance cheese firmness during the cheese-making process. This research uniquely compared calcium chloride extraction from chicken, duck, and ostrich eggshells using hydrochloric acid, a rarely explored approach in the literature. It examined various drying methods—oven-drying, spray-drying, and freeze-drying—to assess their effects on yield and properties, optimizing production processes for efficiency. The findings highlighted the potential to transform eggshell waste into a high-value calcium-rich food additive, addressing waste management while providing a sustainable nutrient source.

2. Materials and methods

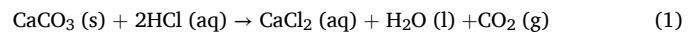
2.1. Materials

Hydrochloric acid with a concentration of 37%, sodium hydroxide

with 97% purity, and calcium chloride in analytical grade were provided (Merck, Darmstadt, Germany). Chicken, duck and ostrich eggshells were collected from the waste of egg production centers.

2.2. Synthesis of CaCl_2

After being collected, the discarded eggshells were cleaned with warm, distilled water, dried for two hours at 120 °C, and then pulverized using a porcelain mortar to a fine powder. The 4% w/v HCl solution was combined with eggshell powder in a 1:20 (w/v) ratio. Mixed solution was sonicated for 5 min to increase the reaction rate and fast releasing carbon dioxide gas (CO_2) (Eq. (1)). Excess acid is neutralized by titration with sodium hydroxide (NaOH) using phenolphthalein as indicator. After that, mixtures were centrifuged at 1774 rpm for 10 min to separate the floating material. After filtering and separating the supernatant, three methods of spray dryer, freeze dryer and oven were used for drying until the liquid was completely evaporated and white calcium chloride crystals were obtained (Garnjanagoonchorn & Changpuak, 2007).



2.3. Drying methods

2.3.1. Oven drying

The calcium chloride samples were spread in a plate and subjected to oven drying at 80 °C for 24 h. After drying, the solid obtained was milled using a laboratory mill (Uni Mill M05-U, UK) to produce a fine powder. Given that a 40-mesh screen was installed, featuring an opening size of approximately 0.42 mm, and considering the milling mechanism of the unit, it is estimated that the resulting powder predominantly has a particle size of <100 µm, which is ideal for ensuring good flowability and for future analyses. The resulting powders were promptly placed in airtight jars to prevent moisture absorption (Jokar et al., 2024; Puspitasari et al., 2019).

2.3.2. Spray drying

To make a slurry, the prepared sample was diluted with water in a 20:80 ratio. To avoid any obstructions in the nozzle, the slurry was passed through an 80-mesh sieve with opening size of approximately 0.177 mm (or 177 µm) as part of the last feedstock preparation process. A steady temperature of 150 °C and a flow rate of 30 ml/min were used to run the pilot-scale spray dryer. The resultant powder, which flowed freely, was promptly placed in zip-lock plastic bags for storage (Puspitasari et al., 2019).

2.3.3. Freeze drying

Using a lab vacuum freeze drier (BEW HAY/SB4, UK), freeze drying was carried out for 96 h at −110 °C and 0.01 Pa of pressure. Prior to freeze-drying, the samples were stored in a deep freezer set to −20 °C for 48 h to lower their temperature. After freeze-drying, the product was milled with a Uni Mill M05-U (UK) using a 40-mesh screen (opening size ~ 0.42 mm). The resulting powder predominantly has a particle size of <100 µm, ensuring good flowability for subsequent analyses (Borhani Esfahani et al., 2023; Puspitasari et al., 2019).

2.4. X-ray fluorescence (XRF)

The spectroscopic method can also be combined with X-ray fluorescence (XRF) to determine the chemical compositions of CaCl_2 powders. For X-ray spectrometry analysis, the Hitachi X-MET8000 Geo portable XRF spectrometer was utilized.

2.5. X-ray diffraction (XRD)

A mortar was used to completely grind the samples until a fine powder was achieved. The analysis was carried out utilizing a Bruker D8 Advanced in a theta-theta configuration with a nickel filter and Cu- α irradiation. Using a rotation speed of 80 rpm and increments of 0.034 degrees, diffraction angles (2θ) 5–60 degrees were examined at a rate of 0.75 s each (Szymanska-Chargot et al., 2018).

2.6. Fourier transform infrared spectroscopy (FTIR)

Utilizing a Nicolet 6700 Fourier transform infrared spectrometer (FTIR, Thermo Scientific, Waltham, MA, USA), FT-IR spectra were captured. The range of the spectra that were gathered was 4000–650 cm^{-1} (Szymanska-Chargot et al., 2018).

2.7. Thermogravimetric analysis (TGA)

A Shimadzu TGA-50 thermobalance was used for TGA. About 8 g of powders were heated in a nitrogen environment between 50 and 950 °C in this investigation at a flow rate of 20 ml/min and a heating rate of 25 °C/min Razali et al. (2022).

2.8. Differential scanning calorimetry (DSC)

Mettler-Toledo's TA4000 differential scanning calorimeter (Greifensee, Switzerland) with a refrigerated cooling unit was used to conduct the DSC analysis. Heat flow was calibrated using 28.45 J/g for the fusion of indium. Temperature calibration was conducted at the melting points of −93.5 °C for hexane, 0.0 °C for water, and 156.6 °C for indium. For a day, a 15–20 mg oleogel was kept at room temperature in 160 ml aluminum DSC pans that were not hermetic sealed. After that, the samples were heated at a rate of 5 °C/min from 20 to 80 °C while being exposed to a nitrogen flow of 20 ml/min. The reference in the DSC cell was an empty pan (Ardiansyah et al., 2023).

2.9. Scanning electron microscopy (SEM)

SEM with an FEI Quanta 200 FEI-ESEM at 1.5 kV under high vacuum conditions was used to examine the powders' topography. Powder samples were mounted in epoxy under vacuum, then ground and polished using standard metallographic methods to provide cross-sectional samples for SEM investigation. After that, an ion sputtering machine was used to sputter an ultrathin coating of gold (about 1 nm) onto each sample (Szymanska-Chargot et al., 2018).

2.10. Chemical analysis

The pH value and ash content of powders were measured according to AOAC methods.

2.11. Yield assessment

The yield of the powders was calculated using Eq. (2) (Chahal et al., 2021).

$$\text{yield (\%)} = \frac{\text{weight of the crystals obtained from drying}}{\text{weight of the crushed eggshell used}} \times 100 \quad (2)$$

2.12. Purity assessment

Spectrophotometry was used to assess the CaCl_2 samples' purity. To put it briefly, 50 ml of distilled water was used to dissolve 1 g of calcium chloride powder, and a vortex was used to fully mix the mixture. At 455 nm, the samples' absorbance was then measured (Bastan et al., 2017).

2.13. Flowability index

Ten grams of the powders were weighed and pushed through a funnel with a height set at 12 mm, allowing them to form a mass on a flat horizontal surface. The angle of repose was determined by measuring the inclination of the mass relative to the base surface. Experimental results indicated that angles between 25–30 degrees correspond to excellent flowability, while angles greater than 66 degrees indicate very poor flowability (Shirkole & Sutar, 2018).

To calculate the flowability index (FI) using the formula:

$$\text{Flowability index} = \frac{10}{\tan(\text{Repose angle})}$$

2.14. Particle size distribution

A laser scattering particle size analyzer (LS 230, Beckman Coulter, USA) was utilized to measure the particle size distribution. Prior to testing, the sample was diluted to <0.02% w/w to minimize multiple scattering effects. The reported particle size of approximately 200 nm refers to the effective size of the particles as measured by the analyzer. It is important to note that laser light scattering methods have limitations due to the diffraction limit, which may affect the accuracy of measurements for particles in the sub-micron range. Consequently, the effective size may include contributions from agglomerates. Sample preparation included efforts to break down these agglomerates to enhance flowability, and the measurements were conducted in duplicate (Padhi & Dwivedi, 2022).

2.15. Color assessment

The color of the powders was determined using a color flex meter (Hunter Association Laboratory, Inc.) set to CIELAB settings (b^* , a^* , and L^*). The findings were reported as Hunter color values: b^* represents yellowness (+) and blueness (−), a^* shows redness (+) and greenness (−), and L^* signifies lightness (+) and darkness (−). The total color change was computed to determine the difference between two colors in the Lab* color space. Eqs. (3) and 4 were used to obtain the whiteness index (WI) and the overall color change (ΔE) (Padhi & Dwivedi, 2022).

$$\text{Whiteness index} = 100 - \sqrt[3]{(100 - L)^2 + a^2 + b^2} \quad (3)$$

$$\Delta E = \sqrt{(L_0 - L)^2 + (a_0 - a)^2 + (b_0 - b)^2} \quad (4)$$

2.16. Statistical analysis

Analysis of variance (ANOVA) was used in a totally randomized design with SPSS software (SPSS Inc., Chicago, IL, USA). The least significant difference (LSD) test was used to get the mean at a 5% significance level.

3. Results and discussion

3.1. Chemical composition analysis

Table 1 presents the X-ray fluorescence spectrometer (XRF) results for chicken, duck, and ostrich eggshells, along with their raw counterparts. The table provides a detailed elemental analysis of various eggshell samples, showing the concentrations of elements such as Chloride (Cl), Calcium (Ca), Phosphorus pentoxide (P_2O_5), Magnesium oxide (MgO), Sodium oxide (Na_2O), Sulfur trioxide (SO_3), Silicon dioxide (SiO_2), Strontium oxide (SrO), Iron(III) oxide (Fe_2O_3), Potassium oxide (K_2O), Copper(II) oxide (CuO), Zinc oxide (ZnO), Titanium dioxide (TiO_2), and Aluminum oxide (Al_2O_3). Ca is the predominant component in all raw eggshells, with concentrations of 98.30% for

Table 1

The X-ray fluorescence spectrometer (XRF) results for the calcium chloride derived from chicken, duck, and ostrich eggshells, as well as the raw eggshell powders.

Sample	Cl	Ca	P ₂ O ₅	CuO	K ₂ O	MgO	Na ₂ O	SO ₃	SiO ₂	SrO	Fe ₂ O ₃	ZnO	TiO ₂	Al ₂ O ₃	Sum
Ostrich-raw	0.024	98.30	0.222	0.051	0.062	0.391	0.130	0.182	0.190	0.060	0.070	0.045	-	0.130	99.857
Chicken-raw	-	97.41	0.247	0.044	0.070	0.720	0.140	0.0304	0.341	0.130	0.068	0.049	-	0.150	99.399
Duck-raw	0.044	93.47	0.558	0.047	0.138	0.859	0.400	0.502	2.24	0.116	0.091	0.037	0.075	1.08	99.657
Ostrich Freeze drying	54.34	44.69	0.201	0.035	0.029	0.140	0.057	0.078	-	0.070	-	-	-	-	99.640
Ostrich spray drying	53.79	45.48	0.043	0.046	0.039	0.130	0.060	0.043	-	0.040	0.063	-	-	-	99.734
Ostrich oven drying	54.59	44.48	0.172	0.039	-	0.140	0.071	0.045	-	0.093	-	-	-	-	99.630
Chicken Freeze drying	54.43	44.67	0.185	0.045	-	0.150	0.056	0.046	-	0.097	-	0.013	-	-	99.692
Chicken spray drying	53.58	45.20	0.410	0.038	0.033	0.261	-	0.110	-	0.053	-	-	-	-	99.685
Chicken oven drying	54.71	44.19	0.150	0.041	0.050	0.414	0.087	0.066	-	0.053	-	-	-	-	99.761
Duck Freeze drying	54.24	44.60	0.282	0.040	0.037	0.160	0.070	0.095	0.100	0.087	0.044	-	-	-	99.755
Duck spray drying	54.04	45.06	0.140	0.037	0.033	0.140	0.052	0.053	-	0.098	-	-	-	-	99.653
Duck oven drying	56.28	42.68	0.150	0.031	0.039	0.324	0.073	0.082	-	0.053	0.047	-	-	-	99.759
Commercial (control)	55.15	43.91	-	0.042	0.025	0.032	0.504	0.068	-	0.018	-	-	-	-	99.749

ostrich, 97.41% for chicken, and 93.47% for duck. After drying, the concentrations of Ca decrease significantly. For instance, ostrich eggshells show a reduction to 44.69% (freeze drying), 45.48% (spray drying), and 44.48% (oven drying). This reduction indicates a transformation of CaCO₃ into CaCl₂, suggesting a considerable loss during the drying process. The increase in Chloride (Cl) content in dried samples is notable, with values rising to 54.34% (freeze drying) for ostrich eggshells, indicating substantial accumulation of Cl in the dried material. The concentrations of other elements, such as P₂O₅, MgO, and SiO₂, vary across samples and drying methods. Phosphorus (P₂O₅) levels are relatively low, with the highest concentration at 0.558% in raw duck samples. The presence of elements like Na₂O and SO₃ is also observed, but generally at lower concentrations compared to Ca and Cl. The different drying methods (freeze, spray, and oven) yield varying elemental compositions. Freeze drying generally retains a slightly higher amount of Ca compared to spray and oven drying. Spray drying shows moderate retention of Ca along with an increase in Cl, while oven drying results in the lowest retention of Ca among the drying methods for duck eggshells. These significant changes in elemental composition highlight the impact of drying conditions on the chemical characteristics of eggshells. The loss of Ca and the gain of Cl are critical for understanding the potential applications of dried eggshells in various fields, such as agriculture and material science. Overall, Table 1 effectively illustrates the chemical transformations occurring in chicken, duck, and ostrich eggshells during the drying processes. The reduction in calcium and the increase in chloride are particularly noteworthy, offering insights into the effects of different drying methods on the material properties of eggshells. Further studies could explore the implications of these changes for their use in various applications. The analysis clearly indicates trends among the three types of eggshells. Ostrich eggshells consistently exhibit the highest initial concentrations of Ca, while duck eggshells show the lowest. After drying, the differences in elemental retention highlight the distinctive physicochemical properties of each eggshell type, suggesting that the choice of drying method can be tailored to optimize specific characteristics for various applications. To complement the chemical analysis, the microstructure of the raw materials should be examined.

To gain a deeper understanding of the physicochemical properties affected by different drying methods, it is essential to examine the underlying mechanisms involved. Each drying technique influences moisture removal pathways differently; for instance, freeze drying typically allows for a more gradual sublimation of water, preserving structural integrity better than oven or spray drying, which can induce more rapid thermal degradation. Additionally, the structural alterations observed in the dried samples are closely linked to the rate of water removal, affecting how tightly bound the remaining components become. The chemical composition changes, particularly the transformation of CaCO₃ to CaCl₂, suggest that variations in drying temperature and atmosphere can lead to differential degradation of calcium content. By elaborating on these processes, we can clarify not only how

drying methods impact elemental retention but also how they influence the overall stability and functionality of the eggshell-derived products in various applications. Such insights will enrich the scientific discussion and provide a clearer perspective on the relationships among drying methodologies, physicochemical transformations, and potential uses.

3.2. XRD patterns

The resulting CaCl₂ powders were subjected to XRD analysis to verify their anhydrous form and purity, as well as to evaluate the homogeneity of the transformation process. The crystallinity of powder internal structures and polymorphism investigations are prominent applications for XRD. The CaCl₂ XRD patterns obtained from eggshells are shown in Fig. 1, which presents the diffraction patterns before and after the acid washing process. The presence of CaCO₃ can be observed at 2θ values of 23.07, 31.52, 36.06, 39.48, 43.26, 47.64, 48.6, 56.62, 57.5, 60.92, 63.2, 64.76, 65.74, 70.29, 72.96, 76.37, 82.15, and 84.9, corresponding to (012), (006), (110), (113), (202), (018), (116), (211), (122), (214), (300), (0012), (0210), (128), (220), (114), (226) crystal planes, respectively. A hexagonal crystal system's reference code, 98-000-5340, and space group R-3c were in accord with the 2θ positions and other characteristics assigned to CaCO₃ by XRD. In the present study, the calcite phase of CaCO₃ formed, with the distinctive peak located at 2θ = 32 and the crystal plane of (104), in good agreement with the findings of Kontoyonis et al. The XRD pattern of the eggshells after the acid washing process, subjected to different drying conditions, showed a strong correlation with dihydrate CaCl₂ (CaCl₂·2H₂O), consistent with the findings of Hussein et al. (2020). The results clearly indicate that all samples exhibited similar XRD patterns, suggesting comparable groups in the calcium chloride powders derived from different eggshell waste. The XRD pattern of the eggshell waste-derived calcium chloride powder reveals an amorphous structure, with characteristic peaks of CaCl₂ observed at several key 2θ values. These results are consistent with earlier research, indicating semi-crystalline characteristics in the material (Moreira et al., 2018). XRD analysis of the obtained CaCl₂ powders confirms that they are primarily dihydrate CaCl₂ (CaCl₂·2H₂O), which crystallizes in the orthorhombic Pnnm space group (Strelec et al., 2024). XRD analysis confirmed that the calcium chloride derived from eggshells is predominantly in the dihydrate form, with peak positions and intensities matching those reported by Reyes-Grajeda et al. (2007). The analysis confirms the high purity and uniformity of the produced calcium chloride dihydrate from wet eggshell waste, as indicated by sharp peaks in the crystalline structure and the absence of secondary phases. A yield of 109 g was obtained from 100 g of eggshell waste using hydrochloric acid treatment. The improved process involved neutralizing excess hydrochloric acid with calcium hydroxide, which was previously omitted. The calcium content measured at 28.37% slightly exceeds the theoretical 27.26%, verifying the dihydrate form of the compound. (Garnjanagoonchorn & Changpuak, 2007). The drying method significantly impacts the crystallinity and purity of CaCl₂ from eggshell waste,

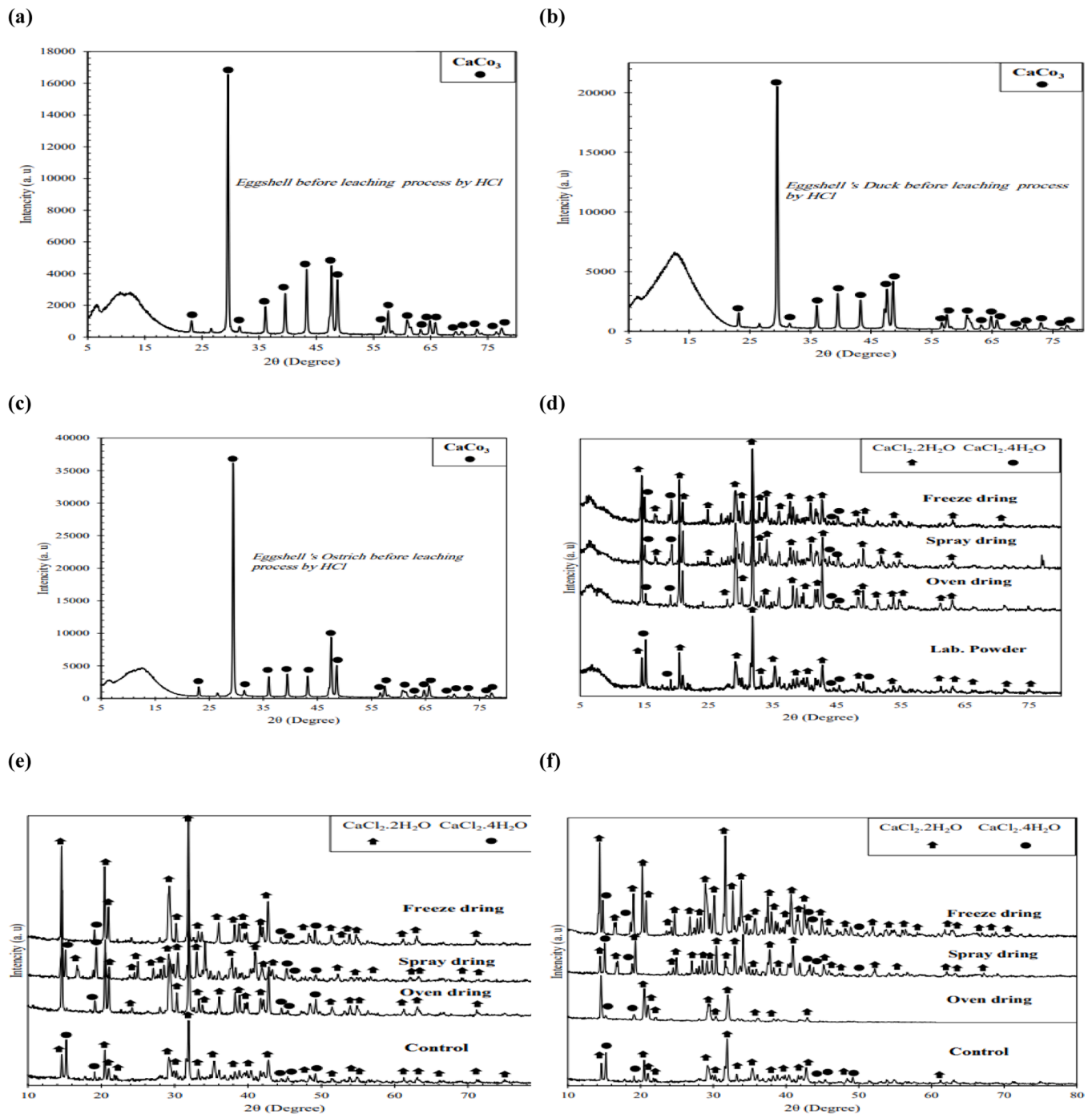


Fig. 1. X-ray diffraction patterns of chicken eggshell (a), duck eggshell (b), ostrich eggshell (c) before the acid washing process with HCl, and chicken eggshell (d), duck eggshell (e), and ostrich eggshell (f) after acid washing process and dried in different conditions.

affecting peak intensities and positions in XRD patterns. Despite variations in drying conditions, XRD results indicate high purity and homogeneity, showing consistent transformation across methods. This understanding is crucial for optimizing calcium chloride production and selecting suitable drying techniques. Analysis reveals that ostrich eggshells have the highest initial calcium carbonate (CaCO_3) content, followed by chicken and duck eggshells. Post-acid washing, all eggshells showed similar XRD patterns indicative of dihydrate CaCl_2 ($\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$). Microstructural analysis revealed differences in porosity and morphology related to chemical compositions and drying methods, which are essential for understanding the physicochemical properties of calcium chloride from eggshell waste.

Elaborating on the mechanisms involved, the drying methods influence the crystallization pathways of calcium chloride derived from eggshell waste, affecting moisture removal efficiency and structural integrity. Freeze drying allows for a gradual and uniform moisture evaporation, which minimizes thermal degradation and preserves the dihydrate form, enhancing crystallinity and purity as indicated by distinct XRD peaks. In contrast, spray drying may lead to rapid evaporation, resulting in less control over crystal growth and potential alterations in the final structural arrangement. Oven drying can induce more significant thermal stress, possibly contributing to variations in crystallinity and increasing amorphous characteristics in the resultant powders. These thermal degradation pathways are crucial for

understanding how each drying technique alters the compositional and structural properties of CaCl_2 , thus affecting its suitability for various applications. Furthermore, the observed variations in porosity and morphology highlight the relationship between drying conditions, chemical composition, and the physicochemical properties of the produced calcium chloride. This insight reinforces the importance of selecting appropriate drying methods to optimize the production and performance of calcium chloride from eggshell waste.

3.3. FTIR spectroscopy

The FTIR spectra of calcium chloride derived from eggshell waste under spray-drying conditions are seen in Fig. 2. All samples exhibit peaks in the $599\text{--}720\text{ cm}^{-1}$ range, which correspond to the Ca–O bond (Rohim et al., 2014). The peaks observed at $2157\text{--}2165\text{ cm}^{-1}$ are associated with the symmetric O–H stretch. Peaks in the range of $1632\text{--}1642\text{ cm}^{-1}$ can be attributed to the H–O–H bending vibration, indicating the presence of water in CaCl_2 . Additionally, peaks at $3521\text{--}3878\text{ cm}^{-1}$ correspond to the asymmetric O–H stretching. The FTIR results indicated that while there were similarities in the functional groups present in the calcium chloride powders derived from different eggshell waste sources, there were also slight variations in the positions and intensities of certain functional groups. These differences may be attributed to the specific types of eggshell waste used in the calcium chloride production. Furthermore, the O–H stretching band is represented by the large peak seen at $3521\text{--}3878\text{ cm}^{-1}$, suggesting that water is present in the crystalline structure of CaCl_2 (Araujo et al., 2021). The formation of the semi-crystalline structure in the eggshell matrix involved intramolecular or intermolecular hydrogen bonding and van der Waals forces (Karunadasa et al., 2018). Several studies using FTIR spectroscopy on calcium oxide derived from eggshell waste indicate the presence of OH groups in Ca(OH)_2 , with peaks around 3600 cm^{-1} and 2360 cm^{-1} . The Ca–O bond is responsible for another band that was seen at 670 cm^{-1} (Rohim et al., 2014). Laouicia et al. (2024) reported that the chemical treatment of *Agave americana* L. leaves with NaOH at varying concentrations and treatment durations had minimal effects on the position and intensity of the peaks. Spray drying may enhance water molecule presence in the crystalline structure, impacting the preservation of functional groups like O–H stretching bands. Understanding these effects is vital for optimizing calcium chloride production, ensuring purity and hydration, and selecting suitable drying methods for desired physicochemical characteristics. FTIR analysis of calcium chloride from chicken, duck, and ostrich eggshells shows similarities in functional groups, particularly in the Ca–O bond and O–H stretching vibrations, though variations in peak positions indicate that the eggshell type influences physicochemical properties. Peaks in the $599\text{--}720\text{ cm}^{-1}$ range reveal consistent calcium bonding, while differences in O–H bands

reflect hydration variations. Additionally, the microstructural characteristics of raw eggshells significantly affect the formation of semi-crystalline calcium chloride, influenced by intramolecular and intermolecular forces such as hydrogen bonding.

Elaborating on the mechanisms at play, the drying methods employed significantly influence the physicochemical properties of calcium chloride extracted from eggshell waste. For instance, spray drying can enhance the incorporation of water molecules into the crystalline structure, as indicated by the prominent O–H stretching bands observed in the FTIR spectra. This presence of water can alter the hydrogen bonding network within the semi-crystalline structure, leading to variations in thermal stability and purity. The differences in O–H stretching band positions and intensities among eggshell types suggest that the specific chemical composition of the eggshells alters the hydration capabilities and therefore the functional characteristics of the resulting calcium chloride. Furthermore, variations in moisture removal pathways during different drying methods can impact thermal degradation patterns and structural integrity. Understanding these underlying mechanisms allows for better optimization of drying techniques to enhance the material properties of calcium chloride, ensuring its suitability for various applications in fields such as agriculture and material science. This deeper insight into the interactions between drying methods and structural characteristics will strengthen the scientific interpretation of the results and inform future research directions.

3.4. TGA and DSC analysis

As organic materials decompose when heated, the mass loss is displayed by the TGA and DSC of the samples (Figs. 3a–b and Table 2). The TGA and DSC analyses indicated an initial weight loss at $260\text{ }^\circ\text{C}$, which corresponds to significant dehydration, reflecting the loss of water of crystallization from the crystal lattice. This process results from the breaking of intramolecular and intermolecular hydrogen bonds. Identify the temperature range ($70\text{ }^\circ\text{C}$ to $160\text{ }^\circ\text{C}$) where the weight remains relatively stable, indicating the robustness of the eggshell-derived calcium chloride's crystal structure under these conditions (Zhao et al., 2022). The structure of calcium chloride made from eggshell waste changed at high temperatures ($200\text{ }^\circ\text{C}$), which resulted in a consistent decrease in weight (Zhang et al., 2019). The TGA curves also confirm the maximum weight loss and the decomposition rate of freeze-dried powders from chicken eggshell waste. DSC analysis is employed to measure the thermal energy released or absorbed during exothermic or endothermic reactions when CaCl_2 is heated. The magnitude and position of the exothermic and endothermic peaks are influenced by the thermal phase transformations of CaCl_2 (Laouicia et al., 2024). The DSC observations of CaCl_2 powders are presented in graphical form in Fig. 3b. According to the findings, the loss or evaporation of absorbed water

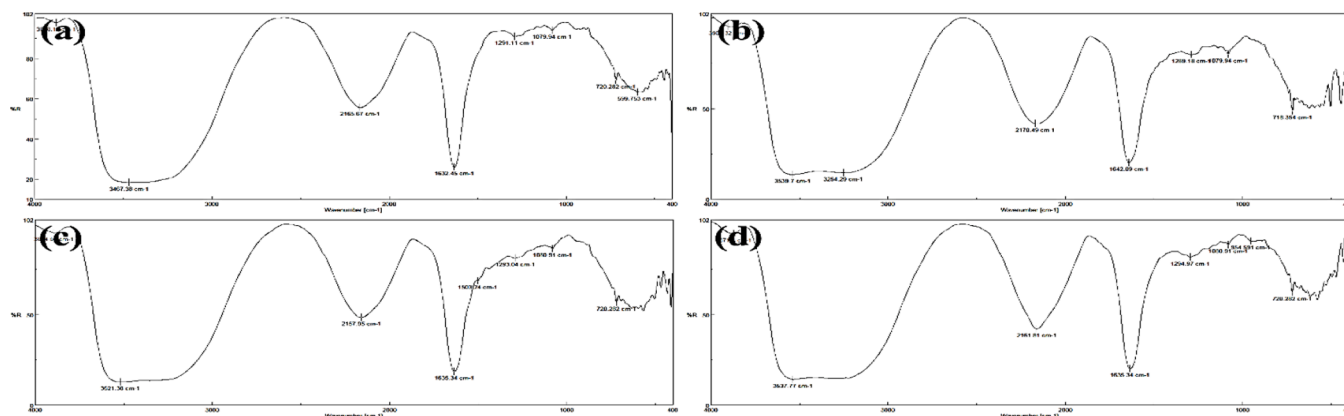


Fig. 2. Fourier transform infrared spectroscopy (FTIR) analysis of eggshell waste derived calcium chloride in respect to spray-drying conditions: ostrich eggshell (a), duck eggshell (b), chicken eggshell (c), and control CaCl_2 (d).

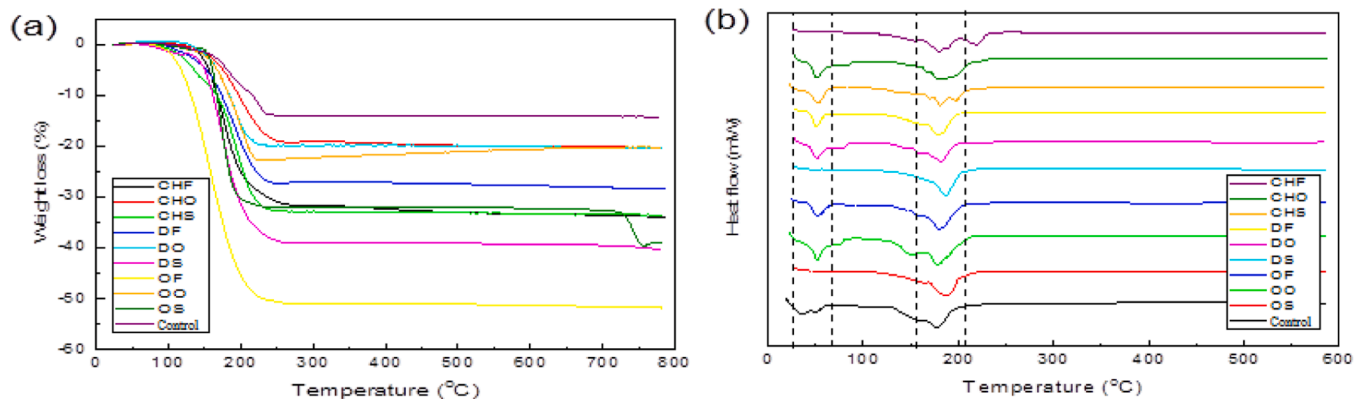


Fig. 3. Thermogravimetric analysis (TGA) (a) and differential scanning calorimetry (DSC) (b) curves of eggshell waste derived calcium chloride. CHF: Chicken eggshell freeze-dried, CHO: Chicken eggshell oven-dried, CHS: Chicken eggshell spray-dried, DF: Duck eggshell freeze-dried, DO: Duck eggshell oven-dried, DS: Duck eggshell spray-dried, OF: Ostrich eggshell freeze-dried, OO: Ostrich eggshell oven-dried, OS: Ostrich eggshell spray-dried.

Table 2
wt loss (%) up to 260 °C.

Sample	CHF	CHO	CHS	DF	DO	DS	OF	OO	OS	Control
Weight loss (%)	31	19.9	32.86	27.3	19.94	38.96	50.86	22.58	32.11	13.98

CHF: Chicken eggshell freeze-dried, CHO: Chicken eggshell oven-dried, CHS: Chicken eggshell spray-dried, DF: Duck eggshell freeze-dried, DO: Duck eggshell oven-dried, DS: Duck eggshell spray-dried, OF: Ostrich eggshell freeze-dried, OO: Ostrich eggshell oven-dried, OS: Ostrich eggshell spray-dried.

corresponds to the first endothermic peak, which happens between 20 and 70 °C. There are very little exothermic or endothermic changes between 70 and 160 °C, indicating that CaCl_2 is thermally stable in this range. At 150–200 °C, another endothermic rise is seen, which is explained by the crystallization water evaporating. The drying method significantly influences the thermal properties of calcium chloride powders, with freeze-dried samples showing higher initial weight loss in TGA due to more effective removal of water of crystallization compared to spray or oven drying. Thermal stability varies by technique, affecting crystal structure integrity, as reflected in TGA and DSC curves. This understanding is crucial for optimizing calcium chloride production and applications, especially when derived from eggshell waste. TGA and DSC analyses of calcium chloride from chicken, duck, and ostrich eggshells reveal trends in thermal stability and mass loss. All samples exhibited initial weight loss at 260 °C, indicating dehydration and hydrogen bond breakdown, but the extent varied, with freeze-dried samples showing greater loss. This suggests that the composition and structure of each eggshell type influence the thermal properties of the derived CaCl_2 . Furthermore, relevant materials from control provide context for the thermal transformations observed in the results. Previous studies emphasize how specific drying techniques affect the thermal stability and integrity of CaCl_2 from organic sources.

To provide a deeper understanding of the mechanisms involved, the effects of different drying methods on the thermal properties of calcium chloride derived from eggshell waste are significant. Each drying technique influences moisture removal pathways, with freeze drying enabling more effective extraction of water of crystallization compared to spray or oven drying. This not only impacts initial weight loss patterns observed in TGA but also contributes to the thermal stability reflected in DSC analyses. For example, the substantial initial weight loss at lower temperatures for freeze-dried samples indicates a more efficient moisture removal, leading to a more stable crystallization environment. In contrast, the thermal degradation pathways alter as drying temperatures increase, affecting the integrity of the crystal structure. Understanding how these mechanisms interact is crucial for optimizing the production and application of calcium chloride, as they dictate the material's thermal stability and compositional integrity across different drying techniques. This nuanced insight enhances our understanding of how

physicochemical properties are influenced by drying methods, ultimately informing more effective application strategies for calcium chloride derived from eggshell waste.

3.5. Microstructure of CaCl_2 powders

The microstructure of the powders was significantly influenced by the drying technique employed. The freeze-dried powders exhibited a polished structure, indicating a more uniform and fine morphology. In contrast, the oven-dried powders displayed substantial alterations to their structure due to exposure to high temperatures. As illustrated in Fig. 4, the morphology of the ostrich spray-dried powder consists of small clusters with spherical shapes, which include both the smallest and largest particle sizes. Fig. 4a–d reveal that the microstructure of ostrich and chicken freeze-dried powders comprises small irregular chunks interspersed with multiple spherical formations. Furthermore, some samples developed multifaceted and angular plate-like structures, as shown in Fig. 4(c, e, f, g, h, and i). The calcium oxide derived from calcined eggshell waste exhibits an uneven morphology, characterized by visible particles of varying sizes, consistent with findings reported by Khalid et al. (2022). The study reveals that drying techniques significantly influence the microstructure of calcium chloride powders derived from eggshell waste. Freeze drying yields a uniform structure, enhancing reactivity and solubility, while oven and spray drying result in notable morphological variations affecting particle shape and size distribution. The microstructure varies by eggshell type and drying method; freeze-dried powders are polished and uniform, whereas oven-dried powders show significant alterations due to high temperatures, leading to uneven sizes. Ostrich eggshells processed via spray drying form small clusters of spherical shapes, indicating variations in particle distribution. This understanding is crucial for optimizing calcium chloride performance in various applications. Relevant materials from control provide context for the observed microstructural changes, emphasizing the importance of selecting suitable drying techniques to enhance the properties of calcium chloride from eggshell waste.

To deepen our understanding of the mechanisms by which different drying methods affect the physicochemical properties of calcium chloride powders, it is important to explore how these techniques influence

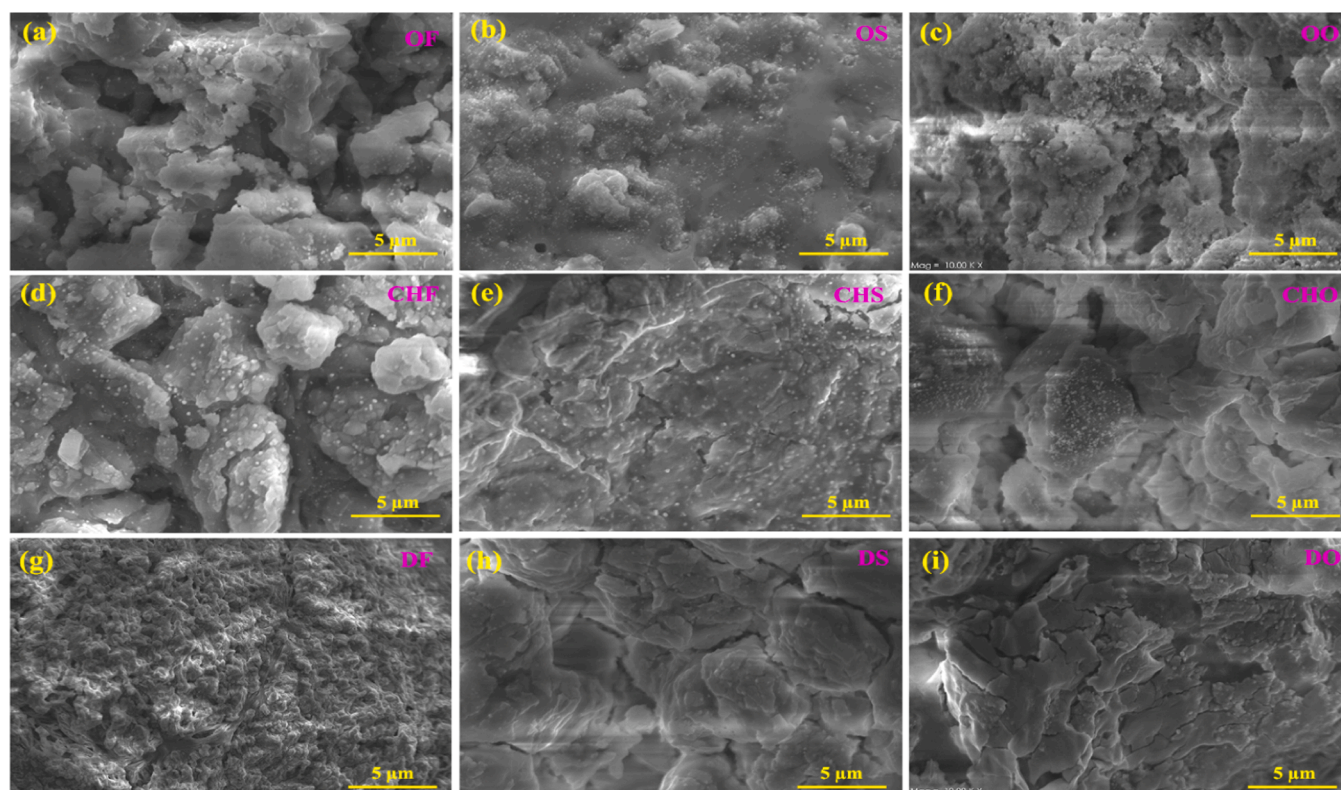


Fig. 4. Scanning electron microscopy (SEM) images of ostrich freeze drying (a), ostrich spray drying (b), ostrich oven drying (c), chicken freeze drying (d), chicken spray drying (e), chicken oven drying (f), duck freeze drying (g), duck spray drying (h), and duck oven drying (i).

moisture removal pathways and resulting microstructural characteristics. Freeze drying promotes efficient moisture evaporation at lower temperatures, which helps maintain a more uniform and polished powder morphology. This structural integrity is crucial as it enhances the reactivity and solubility of the final product. Conversely, the high temperatures associated with oven drying can lead to significant thermal degradation and structural alterations, resulting in uneven particle sizes and shapes. Spray drying, while effective, produces small clusters of spherical particles that may exhibit varied size distributions. Understanding these differences is essential for optimizing the performance of calcium chloride in various applications. Furthermore, the microstructure's variation, influenced by both the drying method and the specific

type of eggshell used, underscores the need for careful selection of processing conditions to tailor the physicochemical properties of calcium chloride derived from eggshell waste for specific uses. This mechanistic insight is critical for improving the overall effectiveness and applicability of the material.

3.6. pH of CaCl_2 powders

The pH of the powders was considerably impacted by the various drying techniques ($p < 0.05$). The control sample had the lowest pH value ($p < 0.05$), whereas the spray-dried CaCl_2 powder had a higher pH value than the oven-dried powder. This difference can affect the

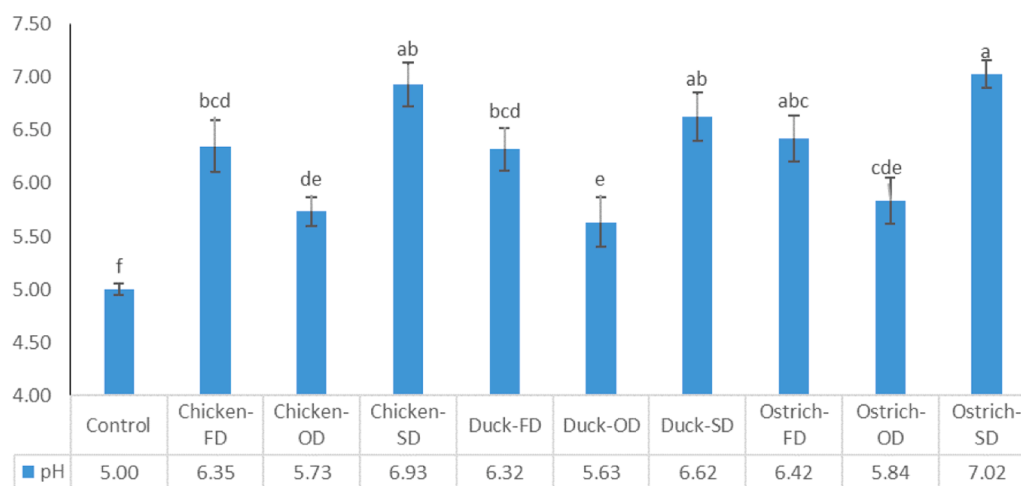


Fig. 5. The pH change of CaCl_2 powder from different drying methods. Freeze drying (FD), oven drying (OD), and spray drying (SD). Values are means $\pm$ SD from three replicates ($n = 3$). Values with different letters show significant difference ($p \leq 0.05$) according to the *Duncan's* new multiple range test.

reactivity and solubility of the powders in various applications. The results of XRF analysis suggest that variations in mineral content, which might lead to disruptions in electrolyte balance, may be the cause of the pH value difference among the CaCl_2 powder samples made using various drying techniques (Cree & Pliya, 2019). The pH value of the CaCl_2 powder in this investigation was unaffected by the kind of CaCl_2 powder extraction source, whether it came from ostrich, duck, or chicken eggs ($p > 0.05$) (Fig. 5). The study examines how drying techniques impact the pH of calcium chloride powders, revealing significant variations. Spray-dried samples exhibit higher pH values, potentially enhancing reactivity for specific applications. The relationship between mineral content and pH is noted, as drying methods influence the electrochemical properties of the powders. Notably, there are no significant pH differences among powders from chicken, duck, and ostrich eggshells ($p > 0.05$), indicating that the drying technique is the primary factor affecting pH. The study found that spray-dried powders have higher pH compared to oven-dried ones, implying that the drying method enhances reactivity and solubility. This consistent pH across different eggshell sources emphasizes the need to control drying techniques for desired physicochemical properties. Variations in pH are linked to mineral content, affecting electrolyte balance and electrochemical properties.

To enhance understanding of the mechanisms influencing the pH of calcium chloride powders derived from eggshell waste, it is essential to consider how different drying methods affect moisture removal and mineral composition. The variations in pH observed among the drying techniques are likely due to differences in how each method impacts the retention and transformation of minerals during processing. For instance, spray drying, which may lead to more rapid evaporation of moisture, seems to result in higher pH values, potentially due to less thermal degradation of sensitive mineral components that can influence reactivity. In contrast, the oven drying process may affect the stability of these minerals more severely, resulting in lower pH values. This nuanced interaction between drying methods and mineral content suggests that the methods utilized not only determine the physicochemical properties but also ultimately influence the electrochemical characteristics of the resulting calcium chloride. Understanding these underlying mechanisms is critical for optimizing the drying techniques to achieve desired pH levels and reactivity for specific applications, reinforcing the importance of controlled processing in tailoring the properties of calcium chloride powders.

3.7. Ash content (%)

The ash content, as indicated in Fig. 6, varies significantly across

drying methods. Oven drying exhibited the highest ash content, suggesting a more extensive conversion of the eggshell material into inorganic salts. Furthermore, the increased dehydration during the oven-drying process is responsible for the higher ash content in the powder that was oven-dried (Cree & Pliya, 2019). The control sample did not show a significant difference in ash content compared to the oven-dried powder ($p > 0.05$). Additionally, there was no significant difference in the amount of ash in the CaCl_2 powder depending on whether it was extracted from ostrich, duck, or chicken eggs ($p > 0.05$) (Fig. 6). The study demonstrates that drying techniques significantly affect the ash content of calcium chloride powders, with oven-drying leading to higher ash levels due to the thermal conversion of organic materials into inorganic salts. This dehydration influences both ash content and the quality and reactivity of the powders, highlighting the need to optimize drying conditions. Importantly, there are no significant differences in ash content among powders derived from chicken, duck, and ostrich eggshells ($p > 0.05$), indicating that the drying method is more critical than the source material. The study confirms that oven drying promotes greater conversion of organic material to inorganic salts due to increased dehydration.

To deepen our understanding of the mechanisms by which different drying methods influence ash content in calcium chloride powders, it is crucial to consider the biochemical transformations occurring during the drying process. Oven drying significantly enhances ash content due to the increased thermal conversion of organic materials into inorganic salts, a process facilitated by higher temperatures that promote dehydration. This extensive conversion implies that moisture removal pathways are critical, as they dictate how effectively organic components are eliminated and, in turn, how much residual inorganic material remains. The uniform ash content across eggshell sources, independent of whether they are derived from chicken, duck, or ostrich, further emphasizes that the drying method plays a more pivotal role in determining ash content than the origin of the raw material. Understanding the interplay between drying techniques and their effects on organic conversion can provide valuable insights for optimizing processing conditions, thereby improving the quality and reactivity of calcium chloride powders in various applications. This mechanistic insight highlights the importance of controlled drying methods in tailoring material properties for specific uses.

3.8. Powder yield (%)

The CaCl_2 powder production from different drying techniques is illustrated in Fig. 7. Oven-drying resulted in the highest powder yield, followed by freeze-drying and spray-drying ($p < 0.05$). This increased

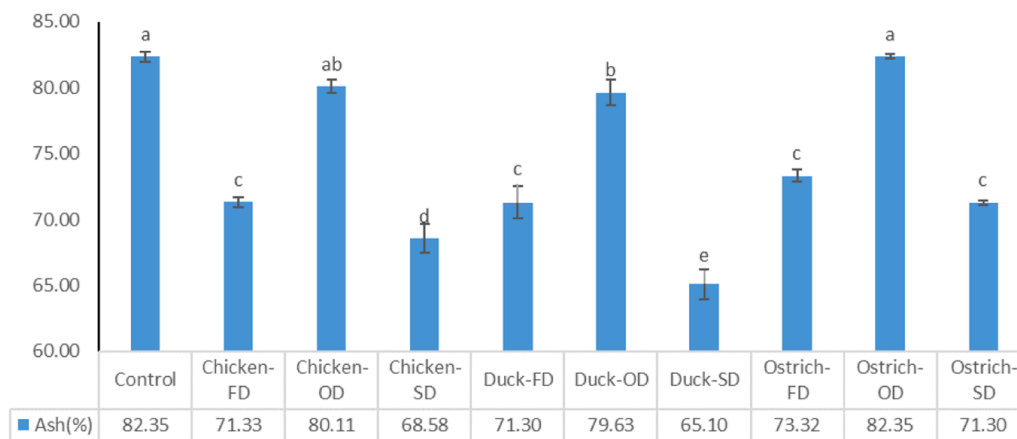


Fig. 6. Ash change of CaCl_2 powder from different drying methods. Freeze drying (FD), oven drying (OD), and spray drying (SD). Values are means $\pm$ SD from three replicates ($n = 3$). Values with different letters show significant difference ($p \leq 0.05$) according to the *Duncan's* new multiple range test.

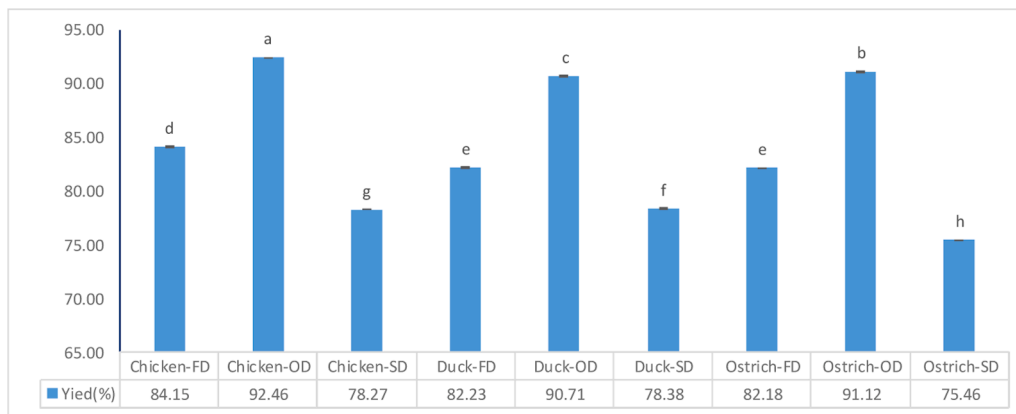


Fig. 7. Powder yield change of CaCl_2 powder from different drying methods. Freeze drying (FD), oven drying (OD), and spray drying (SD). Values are means $\pm$ SD from three replicates ($n = 3$). Values with different letters show significant difference ($p \leq 0.05$) according to the *Duncan's* new multiple range test.

yield can be attributed to the efficiency of moisture removal in oven drying, which requires fewer drying processes and produces a granular powder with improved flowability.

In contrast, freeze-drying showed lower yields due to inherent characteristics such as significant porosity and lower apparent density, which often trap moisture (Argyropoulos et al., 2011). Similarly, poor yields from spray-drying are linked to powder adhering to the drying chamber walls, a problem that worsens with prolonged drying time. Factors such as feed flow rates during spray drying can also contribute to incomplete drying and particle deposition on the chamber walls (Chahal et al., 2021).

Notably, the yield from chicken oven-dried powder was superior to that of other sources ($p < 0.05$), correlating with XRF results indicating the highest total mineral content from chicken eggshells (Table 1). This study underscores the significant impact of drying techniques on CaCl_2 yield, with oven drying proving most effective. The variations in yield are associated with the physical characteristics of the powders, such as porosity in freeze-dried powders and stickiness in spray-dried ones. The findings reinforce the relationship between drying methods, mineral content, and powder yield, highlighting the importance of selecting optimal egg sources for enhancing CaCl_2 production efficiency.

To enhance the understanding of how different drying methods influence powder yield in calcium chloride production, it is essential to delve into the underlying mechanisms that impact moisture removal pathways and structural characteristics. Oven drying achieves higher yields primarily through effective moisture evaporation and reduced process time, allowing for more controlled thermal energy application that promotes granular powder formation. In contrast, freeze drying, while preserving product integrity, often results in lower yields due to significant porosity and lower apparent density that can trap residual moisture, hampering the complete removal of water. Additionally, spray drying frequently encounters challenges such as powder sticking to chamber walls, which exacerbates losses linked to prolonged drying times. The interplay between these drying techniques and their thermal effects can also influence the structural integrity of the resulting powders. Understanding these mechanisms is crucial for optimizing drying parameters to enhance production efficiency and maximize yield. Variations across eggshell sources, particularly the higher mineral content in chicken eggshells, further illustrate the importance of material selection in conjunction with drying methods to achieve optimal CaCl_2 yields. This mechanistic insight provides a more comprehensive understanding of the factors influencing powder yield and informs future strategies for improving calcium chloride production from eggshell waste.

3.9. Flowability index

The flowability index is crucial for the effective production of food-grade materials. It is influenced by factors such as bulk density, particle shape, and size (Al-Ali et al., 2021). As shown in Fig. 8, spray-dried powders and the control sample exhibited the highest flowability values ($p < 0.05$). In contrast, the flowability index of CaCl_2 powders was not significantly affected by oven-drying or freeze-drying methods ($p > 0.05$). Research indicates that spray-dried powders may have more accessible hydrogen bonding sites for water adsorption, contributing to improved flowability (Caliskan & Dirim, 2016). However, this contrasts with findings by Schiano et al. (2018), who suggested that smaller particle sizes increase surface area and cohesive forces, leading to lower flowability. Additionally, Al-Ali et al. (2021) noted that drying processes can alter the textural characteristics of granules, affecting flowability. This study found that the extraction source of CaCl_2 (ostrich, duck, or chicken eggs) had no significant impact on flowability ($p > 0.05$). The drying technique significantly influences the flowability index of food-grade powders, with different methods yielding varying results. Oven drying typically produces larger particles, enhancing flowability, although excessive heat can adversely affect powder structure. Freeze drying results in smaller, porous particles that may increase cohesiveness and decrease flowability. In contrast, spray drying generally achieves the highest flowability by producing spherical particles with a uniform size and accessible hydrogen bonding sites. The flowability index of calcium chloride powders derived from chicken, duck, and ostrich eggshells shows no significant differences based on the eggs' source ($p > 0.05$). However, the study indicates that drying techniques significantly impact flowability, with spray-dried powders and the control sample exhibiting the highest flowability values ($p < 0.05$). This suggests that the drying method is more critical than the type of eggshell in determining flowability.

To provide a more comprehensive understanding of how different drying methods affect the flowability index of calcium chloride powders, it is crucial to investigate the underlying mechanisms that contribute to these variations. The flowability of food-grade materials is significantly influenced by factors such as bulk density, particle size, and shape. For instance, spray drying tends to produce spherical, uniform particles with accessible hydrogen bonding sites, enhancing flowability and making processing easier. This contrasts with freeze drying, which may yield smaller, more porous particles that can increase cohesiveness, thereby reducing flowability. Furthermore, oven drying can create larger particles; while this might improve flowability, excessive heat may lead to structural degradation that adversely impacts texture and cohesiveness. The consistent flowability across different eggshell sources suggests that the drying method itself plays a more pivotal role in determining flowability.

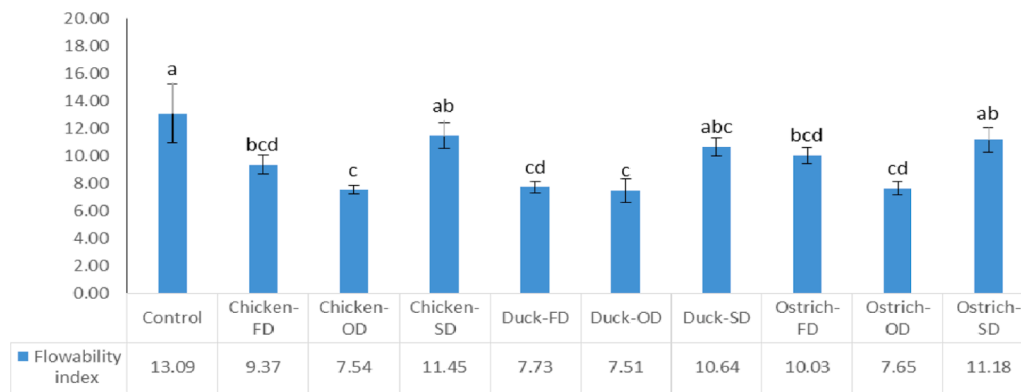


Fig. 8. Flowability change of CaCl_2 powder from different drying methods. Freeze drying (FD), oven drying (OD), and spray drying (SD). Values are means $\pm$ SD from three replicates ($n = 3$). Values with different letters show significant difference ($p \leq 0.05$) according to the *Duncan's* new multiple range test.

characteristics than the type of raw material used. Understanding these mechanistic insights into how drying techniques influence both the physical characteristics and flowability of calcium chloride powders can guide the selection of optimal processing conditions for specific applications in food-grade materials and beyond.

3.10. Purity

According to the findings in Fig. 9, the powders that were freeze-dried and spray-dried had greater purity values than the powder that was oven-dried ($p < 0.05$). Comparing the control sample to the CaCl_2 powders dried using spray and freeze-drying techniques revealed no discernible variation in purity ($p > 0.05$). In the oven-drying method, samples are exposed to high temperatures for extended periods, which may alter the nature of some minerals or cause them to escape and vaporize, ultimately resulting in the formation of different compounds. Decrease in purity of oven-dried powders might be due to changed structure and formed new compounds when using too high extraction temperature and long time (Maroof et al., 2023). It was observed that the type of CaCl_2 powder extraction source—whether from chicken, duck, or ostrich eggs—did not have a significant influence on the purity value of the CaCl_2 powder ($p > 0.05$). The findings reveal that drying techniques notably influence the purity of CaCl_2 powders, with freeze-drying and spray-drying yielding higher purity compared to traditional oven drying. The study underscores the detrimental effects of prolonged high temperatures from oven drying, emphasizing the need for optimized drying conditions to maintain the purity and integrity of the minerals. Importantly, the research indicates that the source of

extraction does not significantly affect purity levels, highlighting that the drying technique is a more critical factor, thereby allowing for sourcing flexibility while achieving desired purity in the final product. The purity of calcium chloride powders derived from chicken, duck, and ostrich eggshells shows no significant differences based on the source of the eggs ($p > 0.05$). This indicates that the extraction source does not substantially affect purity levels. However, the study demonstrates that drying techniques significantly influence purity, with freeze-dried and spray-dried powders exhibiting higher purity values compared to oven-dried powders ($p < 0.05$). Prolonged high temperatures during oven drying can alter mineral structures and lead to the formation of new compounds, thereby decreasing purity.

To comprehensively understand how different drying methods influence the purity of calcium chloride powders derived from eggshell waste, it is essential to consider the mechanistic factors involved in each technique. Freeze drying and spray drying are advantageous as they maintain higher purity levels, primarily due to their lower operating temperatures and faster moisture removal rates, which minimize alteration of mineral structures. In contrast, the prolonged exposure to elevated temperatures during oven drying can lead to thermal degradation, causing some minerals to vaporize or transform into different compounds. This structural alteration not only decreases the purity of the resulting powder but may also introduce unwanted impurities, highlighting the importance of optimizing drying conditions to preserve the chemical integrity of the minerals. Furthermore, the lack of significant variation in purity across different extraction sources—such as chicken, duck, or ostrich eggs—underscores that the drying method itself plays a more critical role than the specific source material.

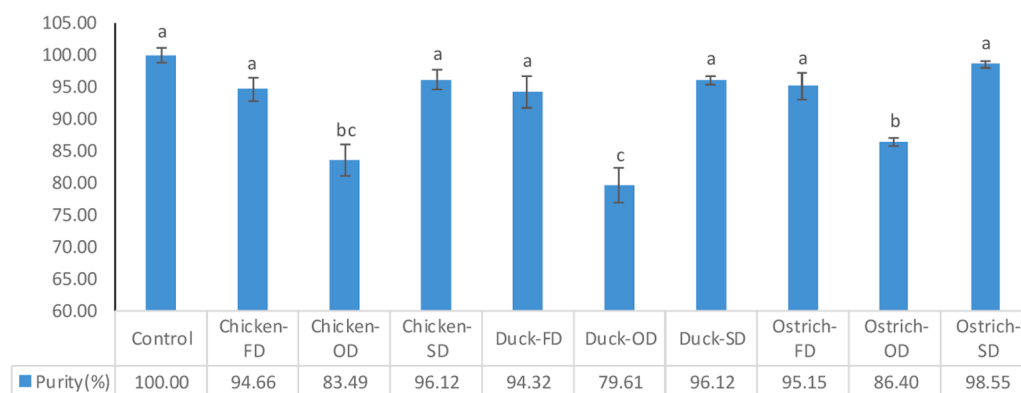


Fig. 9. Purity change of CaCl_2 powder from different drying methods. Freeze drying (FD), oven drying (OD), and spray drying (SD). Values are means $\pm$ SD from three replicates ($n = 3$). Values with different letters show significant difference ($p \leq 0.05$) according to the *Duncan's* new multiple range test.

Understanding these mechanisms enhances our ability to ensure the purity and functionality of calcium chloride powders for various applications, providing insights into optimal processing strategies that can accommodate flexibility in raw material sourcing while achieving desired purity standards.

3.11. Particle size distribution

The drying techniques significantly influenced the particle size of the CaCl₂ powders, as detailed in Table 3. Specifically, the spray-dried powders exhibited a smaller average particle size compared to those that were freeze-dried and oven-dried. This reduction in particle size can be attributed to moisture content; generally, higher moisture content results in larger particle sizes (Roongruangsri & Bronlund, 2016). Since the final product from the spray drying method typically retains lower moisture content than powders produced by freeze drying and oven drying, this factor contributes to the observed decrease in the particle size of the spray-dried powder. Supporting this observation, Xin et al. (2022) reported that the larger particle size of freeze-dried powders could be linked to their poorer grindability index. Additionally, research by Hariadi et al. (2020) indicated that the spray drying method yielded the smallest particle size for anthocyanin pigment powders, while freeze drying resulted in the largest particle size. The study emphasizes the significant influence of drying techniques on the particle size of CaCl₂ powders, revealing that spray drying produces the finest particles. This selection is crucial for tailoring particle characteristics for various applications. Additionally, it highlights the critical relationship between moisture content and particle size, noting that lower moisture levels in spray-dried powders likely facilitate smaller particle formation, enhancing performance. Furthermore, the findings align with previous research by Xin et al. (2022) and Hariadi et al. (2020), reinforcing the validity of the results and their implications on powder characteristics. The particle size distribution of calcium chloride powders derived from chicken, duck, and ostrich eggshells shows that drying techniques significantly affect particle size. Specifically, spray-dried powders exhibited the smallest average particle sizes compared to those produced by freeze-drying and oven-drying. This trend suggests that the drying method is a critical factor in determining particle size rather than the source of the eggshells, which did not show significant differences in particle size ($p > 0.05$). Lower moisture content in spray-dried powders contributes to the formation of finer particles, enhancing their characteristics for various applications. Relevant materials from control have been referenced to support the findings, reinforcing the connection between moisture levels, drying techniques, and particle size distribution.

To deepen our understanding of how different drying methods influence particle size distribution in calcium chloride powders, it is essential to consider the underlying mechanisms involved in moisture removal and structural alteration. The spray drying method yields smaller average particle sizes compared to freeze drying and oven drying, primarily due to its ability to lower moisture content more effectively. In this context, reduced moisture levels facilitate finer particle

formation, as higher moisture content is commonly associated with larger particle sizes, likely due to agglomeration during the drying process. Conversely, freeze drying tends to produce larger particles due to its slower moisture removal rates, which can lead to increased aggregation and poorer grindability. The influence of drying techniques on particle size is crucial for tailoring the characteristics of the powders for specific applications, emphasizing that the drying method has a more significant impact than the source of the eggshell material. This mechanistic insight into the relationship between moisture content, drying methods, and resultant particle size distribution not only reinforces the findings of previous studies but also provides guidance for optimizing drying processes to achieve desired particle characteristics for enhancing performance in various applications.

3.12. Color properties

Color is a crucial sensory component of food goods since it conveys the food's flavor and safety for ingestion (Xu et al., 2023). The color attributes (WI and ΔE) of the CaCl₂ powders are shown to be influenced by various drying techniques and processing conditions, as seen in Fig. 10. It is shown that, oven-dried CaCl₂ powder exhibit a lower and higher WI and ΔE compared to the freeze and spray-dried CaCl₂ powders ($p < 0.05$). This is attributed to the larger particle size of the oven-dried CaCl₂ powder compared to the spray-dried and freeze-dried powders, as particle size and size distribution can influence light reflection and color intensity. A closer examination of Fig. 10 also reveals that the type of CaCl₂ powder extraction source—whether from chicken, duck, or ostrich eggs—had no significant effect on the whiteness index (WI) and total color change (ΔE) of the CaCl₂ powders ($p > 0.05$). All dried powders' ΔE values were substantially higher ($p < 0.05$) than Control's, which may have been caused by a chemical reaction that occurred during the heating process (Fig. 10). When compared to oven-dried powder, the ΔE value was lowest for the freeze- and spray-dried powders. These findings may be explained by the fact that smaller particle sizes in samples with less darkening changed the density, porous surface, and particle characteristics over time, which in turn increased light scattering (Bekele & Emire, 2023). Additionally, according to Suharta et al. (2021), freeze drying produced the greatest drying quality in terms of the color of the andaliman-dried samples. Zia and Alibas (2021) also demonstrated that the cornelian cherry darkened as a result of the extended drying time in the microwave and convective drying, which caused pigment loss. The drying techniques employed have a notable impact on the color properties of CaCl₂ powders. Specifically, oven-dried powders exhibited lower WI and higher ΔE values, indicating that the choice of drying method is crucial for maintaining desirable color characteristics. Additionally, there is a significant correlation between particle size and color intensity, with larger particles in oven-dried powders resulting in decreased light reflection, adversely affecting visual quality. Furthermore, the consistency in color properties across different sources of extraction implies that the drying technique plays a more vital role than the type of eggshell utilized, offering insights into optimizing drying methods for improved and consistent color attributes. The color properties of calcium chloride powders derived from chicken, duck, and ostrich eggshells show no significant differences based on the source of the eggs ($p > 0.05$). Instead, the drying technique has a more pronounced effect on the WI and ΔE. Specifically, oven-dried powders exhibited lower WI and higher ΔE values compared to freeze-dried and spray-dried powders ($p < 0.05$). This trend suggests that the method of drying is a critical factor in maintaining desirable color characteristics. Larger particle sizes in oven-dried powders lead to decreased light reflection and visual quality, as smaller particles in freeze-dried and spray-dried powders enhance light scattering.

To enhance our understanding of how drying methods influence the color properties of calcium chloride powders, it is essential to examine the underlying mechanisms governing these changes. The color attributes, specifically the WI and ΔE, are significantly affected by particle

Table 3
Particle size of different CaCl₂ powder dried.

Sample	Particle size (nm)
Control	270±0.58
Ostrich-OD	322±1.53
Ostrich-FD	273±1.53
Ostrich-SD	231±1.00
Duck-OD	321±0.58
Duck-FD	275±1.53
Duck-SD	231±0.58
Chicken-OD	322±0.58
Chicken-FD	272±1.53
Chicken-SD	231±1.53

Freeze dried (FD), oven dried (OD), and spray dried (SD).

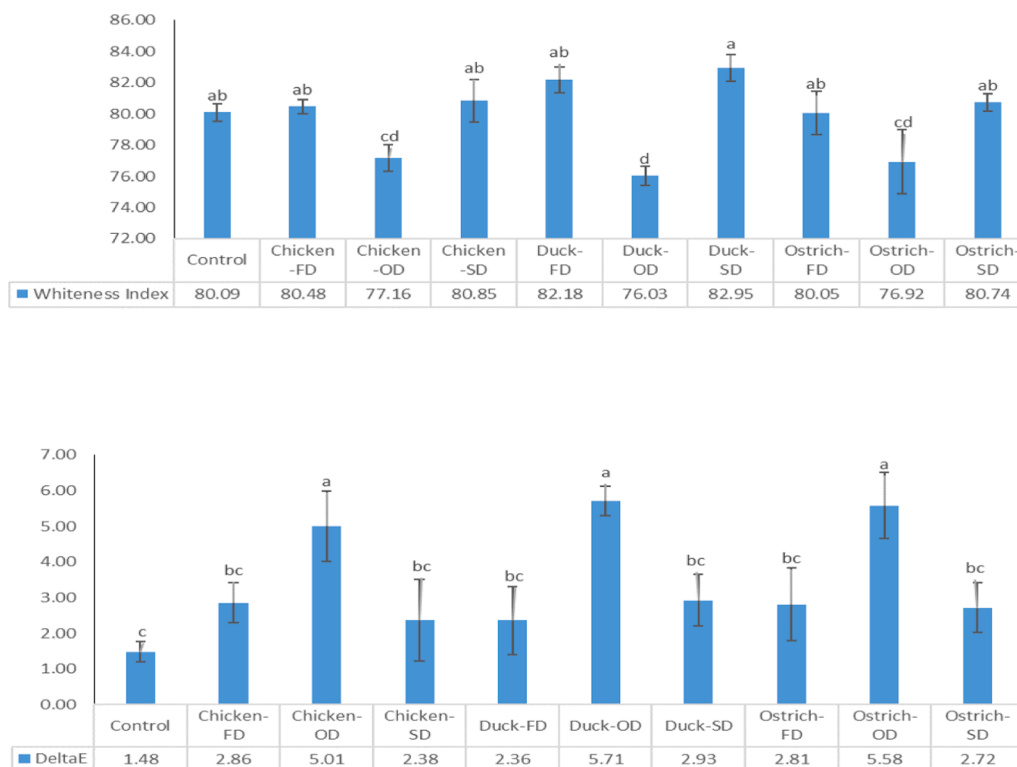


Fig. 10. Whiteness index (WI) and total color difference (ΔE) of CaCl_2 powder from different drying methods. Freeze drying (FD), oven drying (OD), and spray drying (SD). Values are means $\pm$ SD from three replicates ($n = 3$). Values with different letters show significant difference ($p \leq 0.05$) according to the *Duncan's* new multiple range test.

size and distribution, which are influenced by the drying technique employed. Oven drying results in larger particle sizes, leading to decreased light reflection and increased ΔE values, indicative of darker coloration. In contrast, spray drying and freeze drying produce finer particles that promote greater light scattering, enhancing visual quality and maintaining desirable color characteristics. This suggests that the preservation of color during drying processes is closely tied to how moisture is removed and how structural alterations occur in the material. Additionally, the consistent color properties across different eggshell sources highlight the dominance of the drying method over the composition of the raw material. Understanding these mechanisms not only elucidates the relationship between drying techniques and aesthetic qualities of calcium chloride powders but also provides critical insights for optimizing processing conditions to achieve desired color attributes in food-grade applications.

4. Conclusions

The study used a variety of drying techniques, such as freeze-drying, spray-drying, and oven-drying, to create calcium chloride (CaCl_2) from the eggshells of chickens, ducks, and ostriches. According to a chemical composition examination, the main constituents were calcium and chloride, which were mostly found as calcium carbonate (CaCO_3). The calcite phase of CaCO_3 was found in the raw eggshells, and its conversion to $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$ following acid washing was verified by XRD analysis. FTIR spectra revealed various organic functional groups, with slight variations among the different types of eggshells. TGA analysis indicated that chicken eggshells obtained by spray-drying contained significant amounts of calcium carbonate, making them particularly suitable for CaCl_2 production, while the other samples demonstrated limited potential. SEM imaging revealed that the CaCl_2 powders exhibited a non-porous and irregular, almost crystalline morphology. Notably, the type of eggshell source—whether chicken, duck, or ostrich—did not

significantly affect the pH, ash content, purity, flowability, whiteness, or color of the final CaCl_2 powder. The findings of this study suggest that various eggshells can serve as a sustainable and cost-effective source of calcium carbonate for producing CaCl_2 , offering an alternative to traditional methods. This highlights the potential for further research and development to optimize the CaCl_2 synthesis process and explore its applications across various industries.

4.1. Future study for cost-effective production

The synthesized calcium chloride (CaCl_2) from various eggshells—chicken, duck, and ostrich—holds significant potential for future applications across industrial sectors, including water treatment, agriculture, construction, food, pharmaceuticals, and chemical production. Utilizing eggshell waste as a source of calcium carbonate for CaCl_2 manufacturing offers both cost-effectiveness and sustainability. To further minimize production and extraction costs, several strategies can be implemented. Establishing partnerships with local farms ensures a consistent supply of eggshells, reducing transportation costs and promoting waste collection through incentive programs. Process optimization, including efficient washing protocols and optimized hydrochloric acid usage, along with the evaluation of energy-efficient drying methods, is essential to lower operational costs and enhance overall efficiency. While methods such as oven, spray, and freeze drying are commonly used, they are energy-intensive, particularly freeze drying, which can be costly. Therefore, exploring alternative drying methods or optimizing existing ones to reduce energy consumption will be a critical consideration in future research. Scaling up production through larger batch processing and automation can leverage economies of scale, while market analyses inform competitive pricing and the targeting of niche markets that value sustainability. Additionally, investing in research and development to refine extraction methods and explore the utilization of by-products will enhance cost-effectiveness.

Collectively, these strategies not only promote the economic feasibility of CaCl_2 production but also align with the principles of a circular economy and sustainable waste management practices. This suggests opportunities for further research to optimize processes and assess the suitability of the produced CaCl_2 for specific applications.

Ethical statement

The authors declare that they do not have any conflict of interest and the study does not involve any human or animal testing.

CRediT authorship contribution statement

Nasrin Jalali: Writing – review & editing, Writing – original draft, Visualization, Validation, Supervision, Software, Resources, Project administration, Methodology, Investigation, Funding acquisition, Formal analysis, Data curation, Conceptualization. **Mohammad Goli:** Writing – review & editing, Writing – original draft, Visualization, Validation, Supervision, Software, Resources, Project administration, Methodology, Investigation, Funding acquisition, Formal analysis, Data curation, Conceptualization. **Nafisehsadat Sheikhan:** Writing – review & editing, Writing – original draft, Visualization, Validation, Supervision, Software, Resources, Project administration, Methodology, Investigation, Funding acquisition, Formal analysis, Data curation, Conceptualization. **Sharifeh Shahi:** Writing – review & editing, Writing – original draft, Visualization, Validation, Supervision, Software, Resources, Project administration, Methodology, Investigation, Funding acquisition, Formal analysis, Data curation, Conceptualization. **Shabnam Kermani:** Writing – review & editing, Writing – original draft, Visualization, Validation, Supervision, Software, Resources, Project administration, Methodology, Investigation, Funding acquisition, Formal analysis, Data curation, Conceptualization.

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

Data will be made available on request.

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Further reading

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