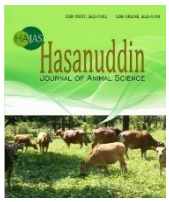

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Calcium Carbonate Content and Proximate Composition of Chicken Eggshell Flour at Different Oven Temperatures

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ABSTRACT

Eggshells, a common household waste product, are rich in calcium carbonate, which constitutes approximately 90% of their composition. The weight of an eggshell typically accounts for 9-12% of the total egg weight. In Indonesia, calcium intake is still relatively low compared to the Recommended Dietary Allowance (RDA) of 1000-1200 mg/day, leading to deficiencies that affect bone health and increase the risk of diseases such as osteoporosis. This study aims to explore the potential of chicken eggshell waste as a valuable source of calcium carbonate, an abundant yet underutilized material. While eggshell waste can contribute to environmental pollution due to microbial activity, its recycling offers a sustainable solution. By converting eggshells into flour, food products can be enriched with calcium. For instance, eggshell flour at a 0.4% concentration has been successfully incorporated into various food products such as baked puddings, ice cream, cakes, muffins, yeast rolls, popovers, and mayonnaise. Studies have shown that this addition significantly increases calcium content without affecting the taste or cooking quality of the food. The primary objective of this research is to produce calcium carbonate from chicken eggshell waste, thus providing a sustainable approach to waste recycling and creating a valuable, nutritious product. The findings of this study could have significant benefits for the community by reducing waste, particularly from chicken eggshells, while transforming it into a beneficial resource for human health.
Keywords: Waste, eggshell, calcium carbonate

INTROODUCTION

The global population is rapidly increasing, particularly in developing countries such as Indonesia, leading to increased food demand. As egg consumption rises among the Indonesian population, the amount of eggshell waste is also increasing. If not effectively managed, this waste can accumulate over time, causing environmental pollution and negatively affecting the safety and comfort of communities near farms [1] [2] [3] [4] [5] [6] [7]. In addition to household waste, eggshells are also generated by egg hatcheries and the food processing industry [8].

Improper management of eggshell waste can exacerbate environmental problems because eggshells decompose slowly under natural conditions. This decomposition releases gases such as ammonia, hydrogen sulfide, and amines, which contribute to unpleasant odors and attract pests and pathogens, including *Escherichia coli* and *Salmonella* [9] [10]. Therefore, the efficient utilization of eggshells is crucial for mitigating their negative environmental impact.

Eggshells are primarily composed of approximately 94% calcium carbonate, with smaller amounts of magnesium carbonate, calcium phosphate, and other organic materials. They also contain essential trace minerals, including phosphorus, magnesium, sodium, potassium, zinc, manganese, iron, and copper [11]. This high mineral content makes eggshells a valuable resource, particularly as a source of calcium, an essential nutrient for bone and dental health, muscle function, hormone secretion, and blood clotting [12].

In Indonesia, calcium intake falls below the recommended dietary allowance (RDA) of 1000-1200 mg/day, contributing to widespread malnutrition and stunting, which are critical national issues. Since the body cannot produce calcium, it must be obtained through dietary sources [11]. The bioavailability of calcium from eggshells is notably high, reaching 93.8%, making them an excellent solution for addressing calcium deficiencies. By processing eggshells into flour or powder, they can be added to food products, increasing their calcium content without altering the taste or cooking quality [13].

Historically, eggshells have been considered waste, primarily generated by food processing industries, including baking, egg flour production, frozen egg manufacturing, large poultry hatcheries, and households. Although small quantities of eggshells have been utilized in various industries, such as animal feed, soil improvement, wastewater treatment, building materials, and even pharmaceuticals [14] [15], most still end up in landfills, contributing to environmental damage [16].

Calcium derived from eggshell flour offers several advantages over synthetic calcium carbonate as it is more easily absorbed by the small intestine [17]. Additionally, it contains beneficial minerals, such as strontium and barium, which enhance its potential as a biomaterial for dietary supplements [18]. Calcium can be efficiently extracted from eggshells using methods such as hydrochloric acid (HCl) treatment [19].

The chemical composition of eggshells consists of 95% calcium carbonate, with the remainder comprising proteins and trace minerals [20]. A typical eggshell weighing 60 g contains approximately 2.3 g of calcium, predominantly in the form of calcium carbonate [21]. The calcium concentration in eggshell powder from white eggs is approximately 113 mg per gram, whereas that from brown eggs is slightly lower at 108 mg per gram [22].

Utilizing eggshell waste in food production offers an eco-friendly approach to reducing waste generated by the egg industry. As eggshells are a rich source of calcium carbonate, this form of calcium can be extracted and used in various food applications [23]. This study aimed to investigate the effects of different oven-heating durations on the calcium carbonate and nutrient levels in chicken eggshells. The goal of this study was to identify the optimal oven time and temperature to maximize both the calcium carbonate and nutrient content in eggshells, thus providing valuable insights into recycling eggshell waste into a commercially viable, nutrient-rich product.

MATERIALS AND METHODS

This study was conducted at the Poultry Meat and Egg Laboratory of the Faculty of Animal Husbandry, Hasanuddin University, Makassar, Indonesia. Chicken eggshells were obtained from JAPFA Hatchery in Maros, Indonesia. Distilled water and standard chemical reagents were used as required for the experiments.

The equipment used in this study included standard laboratory glassware, a 60-mesh sieve, a Philips® blender, Petri dishes, a Buchner funnel, scissors, a hot plate (Model HP 10-2), filter paper, a mortar and pestle, an oven, spatulas, and glass jars.

A Completely Randomized Design (CRD) was employed for the experimental setup, incorporating a factorial arrangement with three replicates. Two factors were tested in this study: temperature and time. The temperature levels tested were 0°C (control), 100°C, and 200°C. The time points evaluated were 0 h (control), 2 h, 4 h, and 6 h. The combination of these factors resulted in 12 treatment groups (3 temperature levels × 4 time points), with each treatment repeated thrice to ensure the reliability of the results.

Research Methods

Chicken eggshells were obtained from PT JAPFA Hatchery in Maros, Indonesia. Initially, the eggshells were thoroughly rinsed under running water to remove dirt or impurities. The thin inner membrane adhering to the shells was carefully peeled off by hand to prevent contamination during the powdering process.

The cleaned eggshells were boiled in distilled water at 100°C for one hour to sterilize the samples and eliminate any potential bacterial contamination. After boiling, the shells were drained to remove any residual water and left to dry at room temperature until their moisture content was significantly reduced.

Once dried, the eggshells were ground into a fine powder using a high-speed blender. To ensure consistency and eliminate larger particles or impurities, the powder was sifted through a 100-mesh sieve.

Finally, the sieved powder was subjected to oven-drying at two different temperatures (100°C and 200°C) for three separate durations (2, 4, and 6 h), following the experimental design. This step was performed to stabilize the powder and optimize its calcium carbonate content. The dried eggshell powder was stored in airtight containers until further analysis.

Testing Methods

Water Content

The crucible was thoroughly cleaned and dried in an oven at 105–110°C for 30 min to remove any moisture. After cooling in a desiccator for 30 minutes to prevent moisture absorption, the empty crucible was weighed using an analytical balance. Approximately 2 g of the sample was accurately weighed and placed in a crucible. The sample was then dried in the oven at 105–110°C for 3 hours.

After drying, the crucible containing the sample was allowed to cool in a desiccator and weighed. The drying and weighing processes were repeated every 30 min until the difference in weight between two consecutive measurements was less than the specified limit, indicating that a constant weight had been achieved. The water content was calculated using the following formula:

$$\text{Water content (\%)} = \frac{W_{\text{initial}} - W_{\text{constant}}}{W_{\text{initial}}} \times 100$$

Where W_{initial} is the initial weight of the sample, and W_{constant} is the final constant weight after drying.

Ash Content

The porcelain crucible was cleaned, dried in an oven at 105–110°C for 30 min, and then cooled in a desiccator. The empty crucible was weighed and recorded. Approximately 2 g of the sample was weighed and placed into the crucible. The crucible containing the sample was then ashed in a furnace at 600°C for 4 h.

After ashing, the crucible was cooled in a desiccator and weighed again. The ash content was calculated using the following formula:

$$\text{Ash content (\%)} = \frac{W_{\text{ash}} - W_{\text{crucible}}}{W_{\text{sample}}} \times 100$$

Where W_{ash} is the weight of the crucible plus ash, W_{crucible} is the weight of the empty crucible, and W_{sample} is the initial weight of the sample.

Protein Content

Protein content was determined using the semi-micro Kjeldahl method. A 1 g sample was weighed and placed in a Kjeldahl flask along with 2 g of catalyst powder ($CuSO_4$: Na_2SO_4 = 1.2:1) and 2.5 mL of concentrated H_2SO_4. The sample was digested until a clear green color appeared.

After cooling, the digest was distilled using a Kjeltec apparatus with the addition of 6 N NaOH solution. The distillate was collected in an Erlenmeyer flask containing 2% H_3BO_3 and titrated with 0.02 N H_2SO_4 until the solution turned purple. The protein content was calculated using the following formula:

$$\text{Protein content (\%)} = \frac{(B - A) \times N \times 14.007 \times 6.25}{C} \times 100$$

where is the blank titration volume (mL), is the sample titration volume (mL), is the normality of H_2SO_4, and is the sample weight (g), respectively.

Fat Content

Fat content was determined using the Soxhlet extraction method. The fat extraction flask was dried at 105°C for 30 min, cooled in a desiccator for 15 min, and then weighed. A 5 g sample was wrapped in filter paper, placed in a fat extraction thimble, covered with fat-free cotton, and inserted into the Soxhlet extractor. Hexane was used as the solvent for fat extraction. The extraction was performed with reflux for a minimum of 5 h until the solvent in the flask became clear.

The solvent was then evaporated by heating the flask in an oven at 105°C for 60 min or until a constant weight was achieved. After cooling in a desiccator for 20–30 min, the flask was weighed again. Fat content was calculated using the following formula:

$$\text{Fat content (\%)} = \frac{W_{\text{final}} - W_{\text{initial}}}{W_{\text{sample}}} \times 100$$

Where W_{final} is the weight of the flask plus fat, W_{initial} is the weight of the empty flask, and W_{sample} is the weight of the sample.

Analysis of Calcium Levels by the AAS Method

A 5 g sample was weighed into a porcelain crucible and charred on a hot plate, then cooled in a desiccator for 30 minutes. The dried sample was ashed in a furnace at 550°C for 3 h and then cooled in a desiccator. The ash was dissolved in 10 mL of a 1:1 nitric acid (HNO_3) and distilled water mixture. The solution was heated to reduce the volume to 5 mL and then filtered into a 25 mL volumetric flask. The filtrate was diluted to volume with distilled water and analyzed for calcium content using Atomic Absorption Spectroscopy (AAS).

Data Analysis

This study employed a Completely Randomized Design (CRD) with a factorial arrangement consisting of two factors, with each treatment performed in triplicate. Data for calcium carbonate and proximate composition were analyzed using One-way Analysis of Variance (ANOVA). When the ANOVA results showed significant differences ($P<0.05$), a post hoc Duncan's Multiple Range Test was conducted using SPSS software to determine the differences between the treatment means.

RESULTS AND DISCUSSIONS

Moisture Content

The moisture content of broiler chicken eggshell flour varied with oven temperature and heating time. After 2 h at 100 °C, the moisture content was approximately 0.39%; after 6 h at the same temperature, it increased to 0.68%. In contrast, at 200°C the moisture content decreased markedly, reaching the lowest value of 0.13% after 6 h. This pattern suggests that at lower temperatures, prolonged heating may cause water vapor to be retained within the matrix due to incomplete evaporation, whereas higher temperatures promote more efficient moisture removal

and result in lower water content over time. Maintaining low moisture content is essential for product stability and shelf life because it helps inhibit microbial growth and deterioration [24]. Similar trends have been reported by Chilek et al. [3], who observed that increasing oven temperature significantly reduced moisture levels in eggshell-based and other flour products, thereby improving their physical quality and storage stability.

Table 1. Effect of Oven Temperature and Baking Time on Moisture, Calcium, Phosphorus, Protein, Fat, and Ash Content of Broiler Chicken Eggshell Flour

Parameter	100°C (2 hours)	100°C (4 hours)	100°C (6 hours)	200°C (2 hours)	200°C (4 hours)	200°C (6 hours)
Moisture Content (%)	0.39±0.01^{a}	0.41±0.01^{b}	0.68±0.01^{c}	0.39±0.01^{a}	0.15±0.00^{b}	0.13±0.00^{c}
Calcium Content (mg/100g)	31.45±0.22^{a}	31.06±0.18^{a}	29.21±0.25^{b}	29.54±0.20^{a}	34.52±0.19^{a}	30.62±0.21^{b}
Phosphorus Content (mg/100g)	0.15±0.03^{a}	0.16±0.04^{a}	0.16±0.01^{a}	0.15±0.027^{a}	0.16±0.04^{a}	0.16±0.04^{a}
Crude Protein (%)	1.45±0.08^{a}	1.50±0.09^{a}	1.60±0.10^{a}	1.20±0.07^{b}	1.55±0.08^{a}	1.80±0.11^{a}
Crude Fat (%)	0.20±0.02^{a}	0.22±0.03a	0.25±0.03^{a}	0.18±0.01^{a}	0.19±0.02^{a}	0.21±0.02^{a}
Ash Content (%)	5.60±0.02^{a}	6.00±0.03^{b}	6.50±0.04^{c}	5.70±0.03^{a}	6.10±0.02^{b}	6.60±0.04^{c}

Different superscripts in the same row denote significant differences between treatments at the 5% significance level ($P<0.05$).

Calcium Content

The calcium content showed a significant increase when heated at 200°C for 4 h, reaching 34.52 mg per 100 g, compared to 31.45 mg per 100 g at 100°C for 2 h. This indicates that higher heat treatment improves the bioavailability of calcium in chicken eggshells by breaking down organic compounds and releasing calcium from the eggshell matrix. These results are consistent with those of Rosnah et al. [17, 25], who found that thermal treatment increases the calcium concentration in eggshell powder by breaking down organic material and freeing calcium ions. Similarly, Chilek et al. [3] reported an increase in calcium content after heat treatment, which they attributed to the enhanced calcium release caused by the thermal decomposition of the shell matrix [18] [26].

Phosphorus Content

The phosphorus content exhibited a slight, statistically insignificant increase with increasing temperatures and longer heating durations. At 100°C for 2 h, the phosphorus concentration was 0.15 mg/100 g, rising marginally to 0.16 mg/100 g after 6 h at 200°C. This change was not statistically significant, suggesting that the phosphorus content remained relatively stable under the tested thermal conditions. This stability is likely due to the inherent chemical properties of phosphorus compounds, which resist degradation at the applied heating temperatures. Park et al. [15] observed similar findings, noting that high-temperature treatments (up to 900°C) did not significantly affect phosphorus levels in chicken eggshells. Similarly, Fritzen and Benetti [6] found that calcined eggshells effectively removed phosphorus from wastewater, suggesting that controlled heating preserves the functionality of phosphorus [16] [27].

Crude Protein

The crude protein increased significantly with both temperature and heating time. For instance, at 100°C for 2 hours, the crude protein was 1.45%, which increased to 1.80% after heating at 200°C for 6 hours. This increase is likely due to protein denaturation caused by heat, which alters the protein structure, making it more detectable during analysis. Jones et al. [12] found that heating leads to protein denaturation, which increases the accessibility of proteins for analysis [14].

Crude Fat

The crude fat remained relatively stable across all treatments. At 100°C for 2 hours, the crude fat was 0.20%, increasing slightly to 0.21% after 6 hours at 200°C. This minimal change was not statistically significant, suggesting that the fat present in chicken eggshells is resistant to thermal treatment under the tested conditions. The stability of the crude fat is likely due to the inherently low fat levels in the eggshells, which experience minimal impact from the applied heating durations and temperatures. This finding is consistent with that of Santos et al. [18], who reported that the low lipid content in mineral-rich by-products, such as eggshells, remains stable during thermal processing. Zhang et al. [28] also highlighted that fat degradation in low-fat matrices is minimal unless they are exposed to very high temperatures or prolonged heating [29].

Ash Content

The ash content increased with higher temperatures and longer heating durations. At 100°C for 2 h, the ash content was 5.60%, which increased to 6.60% after heating at 200°C for 6 h. This suggests that higher temperatures and longer durations effectively remove organic components and concentrate the mineral content. Setiawan et al. [19] reported similar results, indicating that higher drying temperatures and longer heating times enhanced the ash content in chicken eggshell flour by promoting the evaporation of water and the decomposition of organic matter [30].

CONCLUSIONS

Based on the results of this study, the combination of a higher roasting temperature (200°C) and longer roasting time was able to reduce the moisture content to very low levels while simultaneously increasing the calcium and ash concentrations in broiler chicken eggshell flour, whereas phosphorus and fat levels remained relatively constant, and protein tended to increase. Thus, eggshell flour produced under these temperature variations exhibits good stability and mineral content, making it highly potential to be utilized as a natural calcium carbonate source for food fortification and supplements, as well as an environmentally friendly waste-utilization strategy that supports the improvement of calcium adequacy in the community.

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AUTHORS' CONTRIBUTIONS

All authors contributed to the conception and design of the study, as well as to the drafting and revision of the manuscript. All authors conducted the experiments, performed the data analysis, and reviewed and approved the final version of the manuscript.

COMPETING INTERESTS

The authors declare that they have no competing interests.

ETHICAL CLEARANCE

The authors obtained ethical clearance for the use of animals and animal-derived products in this research, whether through direct contact with animals or the utilization of their products.

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