Formulation of Calcium Microcapsules from Eggshells Using Maltodextrin and Gelatin as Coating Materials for Health Supplements

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ABSTRACT

Eggshell waste is a primary environmental concern resulting from the high consumption of chicken eggs in Indonesia. In 2023, egg consumption reached 6.69 kg/capita/year, producing an estimated 1.86 million tons of eggshell waste. This study investigates, for the first time, the combination of maltodextrin and gelatin via the thin-layer drying method for microencapsulating calcium extracted from eggshells. Eggshells contain around 98% calcium carbonate ($CaCO_3$) and 28% elemental calcium, highlighting their potential as an alternative, low-cost, and sustainable source of natural calcium supplements. However, calcium extracted from eggshells is highly susceptible to environmental degradation, limiting its direct application. To overcome this, microencapsulation technology is applied to enhance calcium stability, solubility, and bioavailability. In this study, calcium was extracted using the Heat Assisted Extraction (HAE) method with 2N HCl at 90°C for 2 hours, then encapsulated with maltodextrin and gelatin at ratios of 1:0, 0:1, 1:1, 2:3, and 3:2. The extracted calcium concentration reached 9.041 mg/g. The best performance was achieved at a maltodextrin-to-gelatin ratio of 3:2, yielding an encapsulation efficiency of 90.33%, a solubility of 77.11%, and a favourable particle morphology. These findings not only demonstrate the potential of eggshell-derived calcium microencapsulation to reduce waste but also contribute to achieving SDG 3 (Good Health and Well-being) by providing alternative calcium sources and to SDG 12 (Responsible Consumption and Production) through waste valorization and sustainable resource utilization.

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1. Introduction

Eggs are one of the most widely consumed food ingredients due to their relatively low price and high nutritional content. According to data from the Central Statistics Agency (BPS), per capita egg consumption in Indonesia in 2023 was 2.212 kilograms (kg) per week. According to data from the National Food Agency [1], chicken egg consumption reached 6.69 kilograms per capita, resulting in a national demand of approximately 1.86 million tons per year. Along with increased consumption, eggshell waste has also increased significantly. According to [2], Indonesia's chicken egg production reached 6,117,905 tons in 2023.

Eggshell waste is often considered valueless and discarded, despite containing approximately 98% calcium carbonate ($CaCO_3$) and 28% calcium [3], making it a potential natural source of calcium for health supplements. In addition to calcium, eggshells also contain magnesium, phosphate, and proteins. The main composition of eggshells consists of 95.1% minerals, 3.3% protein, and 1.6% water, dominated by 98.34% calcium carbonate, 0.84% magnesium carbonate, and 0.75% calcium phosphate [4]. Eggshell calcium has a high bioavailability of 93.80%, making it helpful in improving bone density, particularly in patients with osteoporosis [5]. Other minerals, such as magnesium, act as cofactors in more than 300 enzymes involved in energy metabolism and nucleic acid synthesis [6].

One technological approach to formulating calcium supplements from eggshells is microencapsulation, a technique that coats active compounds with protective materials to form microscopic particles [7]. Microencapsulation functions to protect the core material from environmental influences, improve stability, control active compound release, and enhance delivery efficiency in the body [8]. In this study, a combination of maltodextrin and gelatin was used as a wall material. Maltodextrin was chosen for its high solubility, low viscosity, strong binding ability, and oxidative stability [9]; [10]. However, maltodextrin has weaknesses in emulsion and film formation [11], making it necessary to combine it with gelatin, which has good emulsification ability and can form a stable delivery system [12]; [13]. Gelatin is biodegradable, non-toxic, and easily cross-linked [14]. It is derived from the partial hydrolysis of collagen [15] and contains 19 amino acids, with glycine (27–35%), proline, and hydroxyproline (20–24%) being the dominant amino acids [13]. Mammalian gelatin exhibits superior encapsulation properties compared to poultry and marine gelatin due to its composition [16]. Gelatin-based coatings also extend product shelf life owing to their antioxidant and antimicrobial properties [17].

Based on previous studies, no research has yet investigated the extraction of calcium from microencapsulated eggshells containing a combination of maltodextrin and gelatin using the thin-layer drying method. The thin-layer drying method is an efficient and straightforward drying technique for microencapsulating sensitive materials, such as minerals. This process involves spreading a mixture of materials and coatings in a thin layer, then drying it to produce a dry, encapsulated powder that preserves the active components without damage. Thin-layer drying is preferred for its lower equipment costs, ease of operation, and suitability for small- to medium-scale production. This research is also relevant to Sustainable Development Goal (SDG) 3 (Good Health and Well-being), as it provides alternative calcium sources to support bone health and prevent osteoporosis, and to SDG 12 (Responsible Consumption and Production), by promoting the valorization of waste and the sustainable use of food by-products.

2. Research Methodology

2.1. Materials

The materials used in this study included chicken eggshells collected from local markets in Semarang, Indonesia. HCl 2N and NaOH were of analytical grade (Merck). Maltodextrin and gelatin were used as encapsulating agents. Distilled water (aquadest) was used throughout the experiments. And The equipment used in this study included beaker glasses of 100 mL, 250 mL, and 500 mL capacity, retort stands and clamps, thermometers, glass stirrers, a hot plate, oil bath, magnetic stirrer, glass bottles, filter papers, glass funnels, volumetric pipettes, drop pipettes with rubber ball filler, spatulas, watch glasses, an analytical balance, drying oven, porcelain crucibles and dishes, a muffle furnace, thin wall glassware, sieve trays of 40 and 80 mesh, centrifuge, homogenizer, mortar and pestle, and Petri dishes. Instrumental analyses were performed using an Atomic Absorption Spectrophotometer (AAS) and a Scanning Electron Microscope (SEM).

2.2. Procedures

1) Eggshell Preparation

Chicken eggshells were washed with running water to remove dirt and impurities. The shells were then boiled in distilled water for 15 minutes, drained, and air-dried overnight on a stainless-steel tray. The shells were dried in an oven at 200°C for 10 minutes. After cooling, the dried shells were ground into a fine powder using a blender and sieved through an 80-mesh sieve. The resulting eggshell powder was stored in an airtight container for subsequent extraction and analysis.

2) Extraction Process

A total of 10 g of eggshell powder was extracted using 2N HCl at 90°C for 2 hours. After extraction, the solution was filtered with filter paper to separate the filtrate from the residue. The filtrate was then neutralized by adding 250 mL of 3N NaOH. The resulting precipitate was washed with distilled water until the pH reached 7. The precipitate was separated by centrifugation and dried in an oven to a constant weight. The precipitate was dried in an oven at 105°C for 4 hours, until it reached a steady weight, as determined by reweighing at 30-minute intervals until the weight change was less than 0.1%. The dried solid was then calcined in a furnace at 600°C to obtain nanosized calcium oxide powder [18], [19], [20].

3) Microencapsulation Process

Microencapsulation was carried out using the thin-layer drying method, modified from [21]. The encapsulation solution was prepared by dissolving maltodextrin and gelatin (in the specified ratio) in 40 mL of distilled water. The mixture was stirred manually with a glass rod, and the total solids concentration of the encapsulation mixture was maintained at approximately 20% (w/v) to ensure optimal viscosity for film formation and drying. Followed by the addition of the eggshell extract powder, and homogenized using a homogenizer at 10,000 rpm. The homogenized mixture was poured into Petri dishes at a thickness of 3 mm. Drying was performed in an oven at 50°C for 12 hours until a dry, easily detachable layer was formed. The dried encapsulated product was ground and sieved through a 40-mesh sieve.

3. Results and Discussion

3.1. Encapsulation Yield

Yield in microencapsulation refers to the percentage of microcapsule powder obtained from the total initial materials used. Yield is an important indicator of process efficiency and formulation effectiveness in entrapping active compounds (such as calcium ions) within the encapsulating matrix [22]. The yield of microencapsulation can be calculated using Equation (1).

$$Yield\ (\%) = \frac{\text{Weight of dried microcapsules obtained}}{\text{Total wight of materials used (core+coating)}} \times 100\% \quad (1)$$

Table 1. Encapsulation Yield

Extract : Coating	Maltodextrin : Gelatin	Encapsulate Mass (g)	Yield (%)
3 : 8	1 : 0	5.1450	93.55
3 : 8	0 : 1	3.9166	71.21
3 : 8	1 : 1	4.8597	88.36
3 : 8	2 : 3	4.7433	86.24
3 : 8	3 : 2	4.9680	90.33

Based on Table 1, the highest yield was obtained with a maltodextrin-to-gelatin ratio of 1:0, at 93.55%. This indicates that using maltodextrin as a single-wall material (Maltodextrin:Gelatin = 1:0) yielded the highest yield among the ratios tested. The higher the proportion of maltodextrin, the greater the yield, because maltodextrin has high water solubility and forms a matrix that dries easily [12]. Maltodextrin contributes to the formation of finer powder and higher yield. Gelatin, on the other hand, enhances the stability of the microencapsulation structure by forming a gel network. Still, excessive use can decrease yield due to difficult-to-dry, sticky agglomerates that adhere to the dryer walls. The combination of maltodextrin and gelatin can provide a balance between high yield and capsule strength/stability. Encapsulation efficiency and yield are also influenced by the proportion of these two wall materials.

3.2. Calcium Content Analysis of Eggshell Powder

The calcium content was analyzed in the powder obtained from chicken eggshell extraction using the AAS method. The measurement was performed at a wavelength of 422.7 nm. Based on the analysis, the calcium content detected in the sample was 9.041 mg/g. This value exceeds the limit of detection (LoD) of 0.000430 mg/g and the limit of quantification (LoQ) of 0.0001434 mg/g, indicating that the results are valid and well quantified. The results of the calcium content analysis of eggshell powder are presented in Table 2. Table 2 shows that the HAE method was effective in extracting

calcium from eggshells. The measured calcium content of 9.041 mg/g indicates that chicken eggshells are a potential source of active calcium for organic waste applications.

Table 2. Calcium Content Analysis of Eggshell Powder

Parameter	Sample Name	Result	Unit
Calcium (Ca)	Sample 1	9.041	mg/g

Notes:
The test results are only valid for the sample received by the laboratory.
LoD for Ca analysis: 0.000043012273 mg/g
LoQ for Ca analysis: 0.000143374242 mg/g

This finding is consistent with [23], which reported that the calcium content in chicken eggshells can reach approximately 380 mg/g, depending on biological factors such as breed, age, feed type, and sample preparation techniques. The difference between the theoretical calcium content reported in the literature and the experimental results in this study may be attributed to several factors, including sample preparation techniques, solvent efficiency during dissolution, filtration effects, and biological factors in the chickens (such as age, feed type, and eggshell thickness). Additionally, the use of nitric acid (HNO_3) as a solvent also affects the solubility of $CaCO_3$ in its ionic form, while residual solids remaining after filtration may reduce the detected concentration.

Nevertheless, the calcium level of 9.041 mg/g obtained in this study provides sufficient evidence that chicken eggshells are suitable as raw material for calcium supplement production, particularly in microcapsule form. By encapsulating calcium derived from eggshells, it can be prepared in a more stable, soluble, and easily consumable form.

3.3. Microcapsule Solubility

The solubility test was performed by dissolving 1 g of the sample in 100 mL of distilled water at 37°C under magnetic stirring (200 rpm) for 30 minutes. The undissolved fraction was filtered, dried at 105°C to constant weight, and the solubility was calculated from the mass difference between the initial sample and the dried residue. The solubility test was conducted to determine the solubility of the released active compounds and to assess whether the resulting microcapsules can be used in food products [24]. The results of the microencapsulation solubility test are presented in Table 3.

Table 3. Microcapsule Solubility Test Results

Extract : Coating	Initial Sample Weight (g)	Filter Paper Weight (Before Drying) (g)	Filter Paper Weight (After Drying) (g)	Maltodextrin : Gelatin	Solubility (%)
3 : 8	1	0.7995	1.0167	1 : 0	78.28
3 : 8	1	0.7949	1.0711	0 : 1	72.38
3 : 8	1	0.7961	1.0322	1 : 1	76.39
3 : 8	1	0.7958	1.0512	2 : 3	74.46
3 : 8	1	0.7983	1.0272	3 : 2	77.11

Based on Table 3, it can be seen that the microcapsule formula with a pure maltodextrin coating (maltodextrin-to-gelatin ratio of 1:0) had the highest solubility result of 78.28%. In comparison, the sample with a maltodextrin-to-gelatin ratio of 0:1 had the lowest solubility (72.38%). The highest solubility was obtained with treatments using pure maltodextrin or formulations with a high maltodextrin proportion in the maltodextrin–gelatin mixture.

This trend is consistent with the findings of [25], who reported that higher maltodextrin concentrations are associated with greater solubility. This is attributed to the properties of maltodextrin, which has high water solubility, low viscosity, and the ability to form a good protective matrix, allowing microencapsulated particles to dissolve more easily in water [26]. Maltodextrin is a polysaccharide carbohydrate with abundant free hydroxyl (–OH) groups. These groups readily interact with water molecules and form hydrogen bonds, thereby facilitating faster, more complete dissolution [27]. Gelatin, in contrast, tends to exhibit lower solubility than maltodextrin because it is a protein that can form denser, more elastic matrix layers, leading to slower dissolution [28]. Furthermore, gelatin protein denaturation at certain temperatures may reduce solubility [29].

The addition of maltodextrin to gelatin formulations enhances the solubility of the microencapsulated product, with solubility ranging from 63% to 91%, depending on the maltodextrin ratio and concentration [30]. A higher proportion of maltodextrin yields powders with more consistent solubility and texture; however, it also results in weaker capsule walls, making microcapsules more prone to breakage or damage during processing, storage, or application in food and pharmaceutical products. Therefore, gelatin is needed to reinforce the microcapsule wall structure and improve mechanical strength and stability during storage and transportation.

3.4. Characteristics of Eggshell Microencapsulation

This test aimed to evaluate the suitability of the final product in terms of texture, odour, and colour, as well as the materials used in the formulation process. The test principle is to assess shape, odour, and colour using sensory evaluation [31].

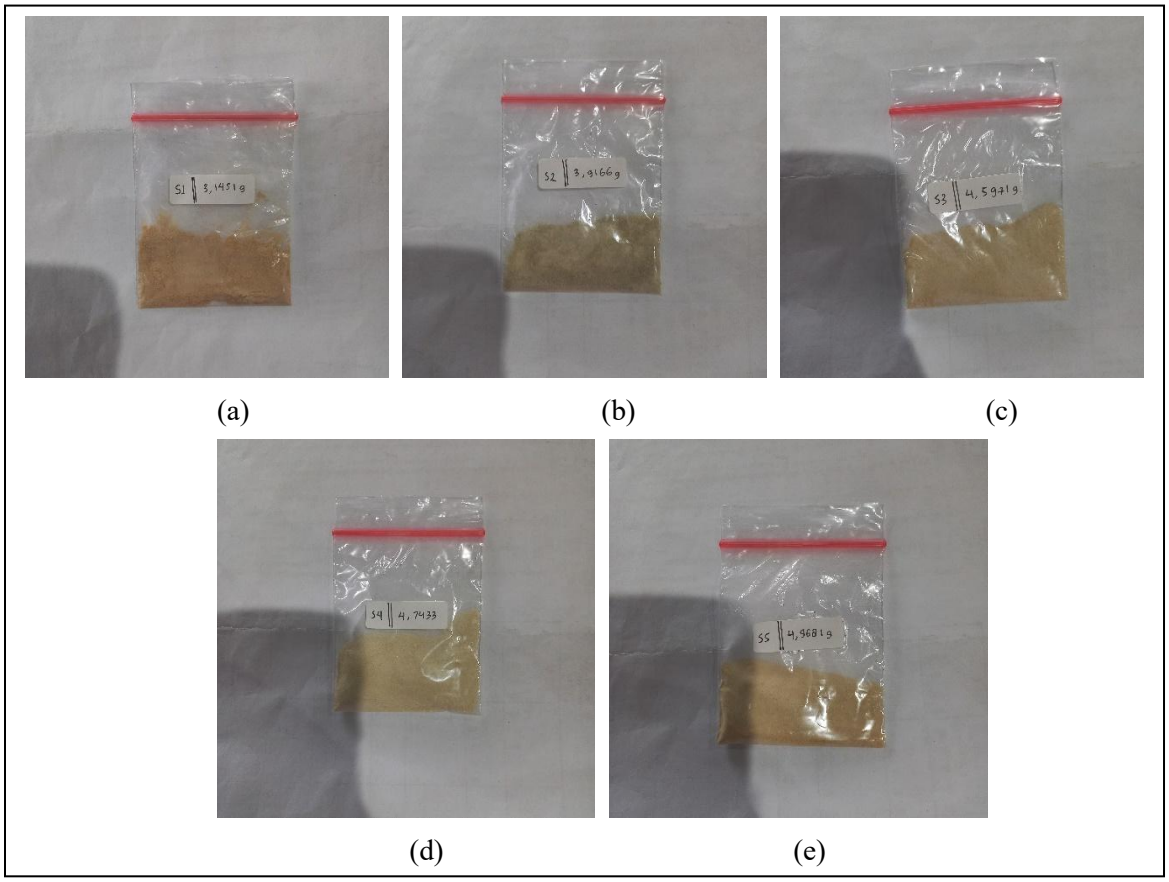

Fig. 1. (a) Encapsulation result of Sample 1 with maltodextrin-to-gelatin ratio (1:0); (b) Encapsulation result of Sample 2 with maltodextrin-to-gelatin ratio (0:1); (c) Encapsulation result of Sample 3 with maltodextrin-to-gelatin ratio (1:1); (d) Encapsulation result of Sample 4 with maltodextrin-to-gelatin ratio (2:3); (e) Encapsulation result of Sample 5 with maltodextrin-to-gelatin ratio (3:2).

Based on Fig.1., it can be observed that Sample 1, with a maltodextrin-to-gelatin ratio of 1:0, had a dark yellow or orange color; Sample 2, with a ratio of 0:1, showed a dark white or greenish color; Sample 3, with a ratio of 1:1, appeared slightly orange-white; Sample 4, with a ratio of 2:3, displayed a white or yellowish color and was the brightest among all samples; while Sample 5, with a ratio of 3:2, showed a slightly orange-white color. Maltodextrin has a yellowish base colour; therefore, microencapsulation using maltodextrin tends to produce darker or orange shades. In contrast, gelatin has a whitish base colour, resulting in brighter microencapsulation. Combining maltodextrin and gelatin in microencapsulation improved the powder's colour quality by enhancing brightness and reducing the intensities of reddish and yellowish hues in the final product.

In organoleptic observation, there were no significant differences in aroma among the five samples. All samples were safe to smell and did not have a pungent odour. The texture comparison of calcium microencapsulation using maltodextrin and gelatin was based on the physical properties and film-forming capabilities of both materials. Samples with higher maltodextrin content exhibited smoother,

non-sticky textures, whereas those with higher gelatin content had rougher, stickier textures. Maltodextrin acts as a filler with low viscosity and good solubility, resulting in microcapsules that are dry, smooth, and easily dissolved. However, maltodextrin has limited emulsifying ability, so its binding capacity for core materials (e.g., oils or calcium) is lower than that of gelatin. Gelatin, on the other hand, acts as an emulsifier, binding core materials more effectively in emulsions, producing microcapsules with denser, chewier, and more stable textures. Gelatin can form stronger matrix layers, contributing to chewy and plastic textures in organoleptic testing. The combination of maltodextrin and gelatin enhances encapsulation efficiency while producing microcapsules with optimal textures that are neither too brittle nor too hard. Excessive proportions of maltodextrin tend to yield brittle, coarse textures, whereas gelatin promotes denser, more elastic textures.

3.5. Morphological Results of Microencapsulation Using Scanning Electron Microscope

Characterization and morphological observation of microencapsulated chicken eggshells were carried out using a Scanning Electron Microscope (SEM) at magnifications of 1000× and 5000×. This technique aimed to evaluate the surface structure, particle shape, and the potential presence of cracks or pores that may have formed during the encapsulation process [32]. SEM observations provide microscopic visual representations of the resulting capsules, thereby facilitating detailed assessment of their morphological quality. This method is widely used in microencapsulation studies due to its ability to produce high-resolution images of particle surface structures [28].

The findings on eggshell microencapsulation using maltodextrin and gelatin as wall materials indicated that the mixture played a crucial role in forming a characteristic particle morphology. The microencapsulation process was carried out with an extract-to-wall material ratio of 3:8 and a maltodextrin-to-gelatin ratio of 3:2. Based on the literature, encapsulation efficiency is influenced by the concentrations of the extract and the coating material. Higher coating concentrations can increase efficiency by forming larger microcapsules, thereby optimising coating capacity. Conversely, increasing extract concentration can decrease efficiency due to limited coating capacity, leaving some extracts unencapsulated. Based on SEM images at 1000× and 5000× magnifications, the microcapsule surfaces appeared relatively dense, with textures that were not entirely smooth. Several particles were observed to adhere to one another, forming agglomerates, and small crystallites were visible on the surface of the capsule. These characteristics suggest that the encapsulation process produced sufficiently compact capsule walls. This is consistent with the findings of [33], who reported that variations in the maltodextrin-to-gelatin ratio led to differences in particle morphology. An unbalanced combination resulted in rougher surfaces, open pores, and less compact microcapsule wall structures.

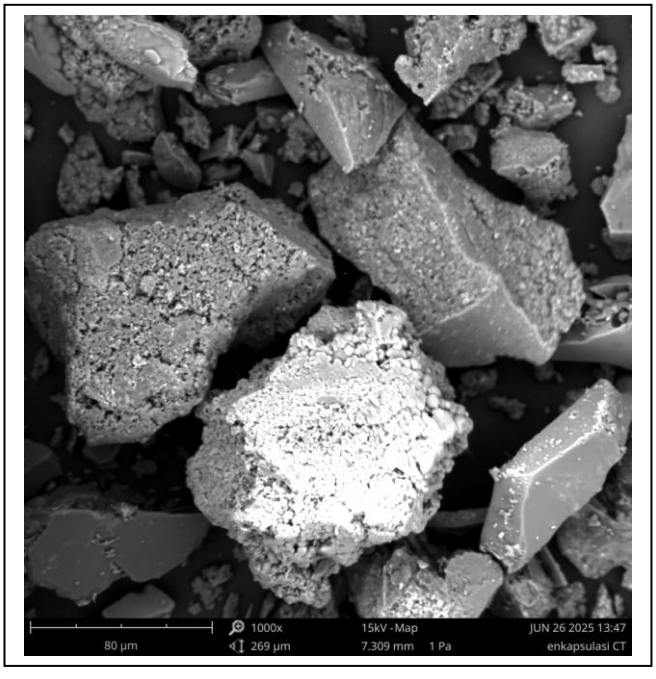

Fig. 2. Morphology of eggshell microencapsulation based on SEM analysis at 1000× magnification

The analysis using SEM in Fig. 2 show that the particle size of eggshell microencapsulation ranges from 10 to 80 micrometres. This size falls into the category of microencapsulation. This classification is consistent with [33], which states that encapsulation can be categorized into three categories based on particle size: macroencapsulation (particle size> 5000 micrometers), microencapsulation (particle size 1–5000 micrometers), and nanoencapsulation (particle size < 1 micrometer). Microencapsulation within this size range is generally considered stable for delivering active compounds. The resulting particle size of the microencapsulated product is influenced by the formulation and process conditions, namely the wall-to-core ratio and the stirring speed. A larger amount of wall material produces a thicker capsule wall, resulting in larger particles, while higher stirring speeds tend to produce smaller particles. Both factors contribute to shaping the overall physical characteristics of microcapsules. This is in line with [34], who reported that the ratio of materials and stirring speed significantly affects the size and morphology of microcapsules.

Based on SEM analysis at 1000× magnification, the eggshell microencapsulation particles appeared irregular, with rough, porous surfaces. The permeable surface suggests that the microencapsulation structure was not entirely homogeneous, possibly due to variations in the ratios of wall materials, such as maltodextrin and gelatin. This finding is consistent with [35], who demonstrated that the wall-to-core material ratio affects the wall structure and porosity of capsules, with higher maltodextrin concentrations resulting in less spherical capsule morphology. The dense structure, with cracks in some areas, indicates that the oven-drying process compromised capsule integrity. This is supported by [36], who reported that higher drying temperatures can damage active compounds, thereby affecting the uniformity of microcapsule morphology. The higher the drying temperature, the more cracks appear on the capsule walls, indicating a weakening of the capsule's physical strength.

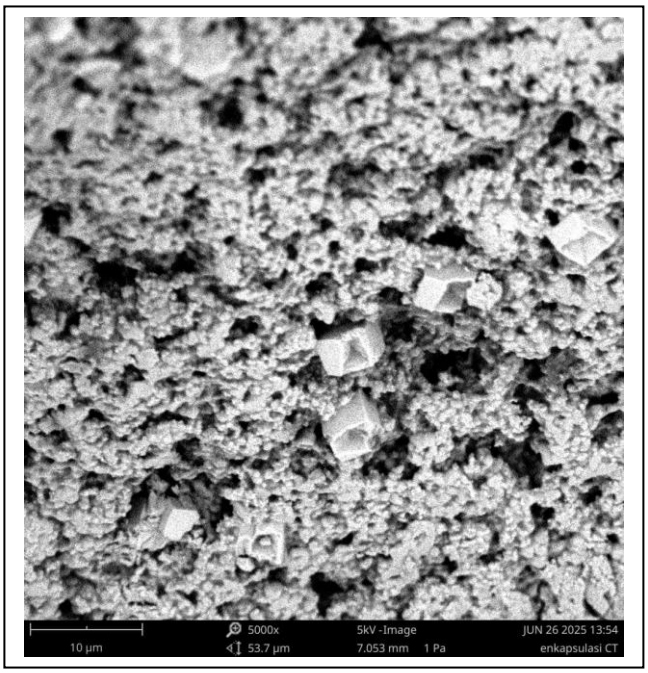

Fig. 3. Morphology of eggshell microencapsulation based on SEM analysis at 5000× magnification

Based on Fig. 3, morphological observation of the microencapsulation using a Scanning Electron Microscope (SEM) at 5000× magnification revealed a more complex surface microstructure with a fine pore distribution and small cube-like crystallites dispersed across several areas. These crystalline surfaces originated from residual calcium minerals in the eggshell that were not completely encapsulated by the coating material. High porosity can increase the specific surface area, which is beneficial for controlled release; however, it may also accelerate the degradation of the active compound if not adequately protected. This finding is consistent with the results of [33], who reported that microcapsules with maltodextrin–gelatin coatings tended to exhibit micropores and residual crystals due to phase incompatibility during drying. The surface morphology of the microencapsulated particles appeared rough with visible wrinkles. Morphologically, most of the particles were not perfectly spherical. Some particles appeared flattened and adhered to one another, indicating that the

capsule wall formation was not yet optimal. Although encapsulation occurred, the resulting microcapsule structures were not fully morphologically stable. The uneven surface and tendency of the particles to wrinkle and stick together were likely caused by shrinkage or internal stress during the drying process. Such conditions are typically caused by rapid evaporation, resulting in uneven heat distribution. These findings are consistent with [37], which reported that indentations on microcapsule surfaces may arise from wrinkles formed by uneven shrinkage during the drying process.

The type of coating material plays a crucial role in determining the morphology of the microcapsules as observed through SEM [38]. The formulation of maltodextrin and gelatin as encapsulating agents influences the formation of capsule wall structures. Maltodextrin is water-soluble, has low viscosity, and possesses film-forming ability, but is brittle. This property tends to produce capsules with uneven, easily wrinkled surfaces, especially during rapid drying [39]. On the other hand, gelatin possesses gelling and film-forming properties, providing elasticity to the capsules [40]. Gelatin helps maintain the integrity of the capsule structure, preventing it from easily breaking or wrinkling during drying [35]. The combination of these two materials in the encapsulation formulation is appropriate, as maltodextrin supports the efficiency of the drying process, while gelatin strengthens the capsule structure [41].

Based on the SEM morphological results, the use of maltodextrin and gelatin resulted in capsules that were not fully compact and displayed fine wrinkles, suggesting that the capsule structure still requires optimisation. Nevertheless, the clearly visible capsule walls indicate that the encapsulation process successfully protected the active compound inside. The porous, slightly rough surface morphology of the microcapsules demonstrates promising potential for dispersion in water and resistance to high temperatures. The microencapsulation of eggshell calcium protects the calcium content from environmental degradation and enhances the stability of the active compound during storage and application. This finding is consistent with Fitri and [42], who reported that the combination of maltodextrin and gelatin is effective for encapsulating active compounds and producing relatively stable microcapsules. However, further optimisation of the ratio and process conditions is required. Considering the physicochemical properties of maltodextrin and gelatin, as well as the ability of the resulting capsules to maintain their shape and wall structure, eggshell microencapsulation using this combination is feasible as a calcium supplement.

4. Conclusion

This study is the first to microencapsulate calcium extracted from chicken eggshells using a maltodextrin–gelatin wall material via thin-layer drying. The calcium extract contained 9.041 mg/g, and the optimal formulation (maltodextrin : gelatin 3:2) achieved an encapsulation efficiency of 90.33% and a solubility of 77.11%. Thin-layer drying produced stable microcapsules with good morphology and uniform particle distribution, demonstrating a low-cost approach for converting eggshell waste into a value-added calcium supplement that supports SDG 3 and SDG 12. Although the highest yield (93.55%) and solubility (78.28%) were obtained using maltodextrin alone (1:0), SEM analysis showed that this ratio produced rough, brittle particles. In contrast, the 3:2 ratio resulted in smoother, more structurally balanced microcapsules—neither too fragile nor too dense—due to the complementary properties of maltodextrin and gelatin. A higher maltodextrin proportion increased surface roughness and brittleness, whereas excess gelatin produced denser, more elastic particles. Overall, the maltodextrin–gelatin 3:2 formulation provided the best combination of efficiency, solubility, and morphology for eggshell-derived calcium microencapsulation.

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