

THE EFFECT OF BLUE SWIMMING CRAB SHELL NANOCALCIUM FLOUR ADDITION ON CALCIUM CONTENT AND PHYSICAL CHARACTERISTICS OF ICE CREAM CONES

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ABSTRACT

Ice cream cones are widely consumed as complementary edible containers for gelato and other ice cream products. Fortification with nanocalcium flour derived from blue swimming crab shells represents a potential strategy for improving the calcium content of ice cream cones while valorizing fisheries by-products. This study aimed to determine the effect of different concentrations of blue swimming crab shell nanocalcium flour on the calcium content and physical characteristics of ice cream cones. A laboratory experimental method was applied using a completely randomized design with four levels of nanocalcium flour addition: 0%, 3%, 6%, and 9%. Parametric data, including moisture content, calcium content, hardness, and resistance to ice cream, were analyzed using analysis of variance (ANOVA), followed by the honestly significant difference (HSD) test when significant differences were detected. Non-parametric data from the hedonic test were analyzed using the Kruskal-Wallis test, followed by the Mann-Whitney test where appropriate. The results showed that nanocalcium flour addition significantly affected ($p < 0.05$) the calcium content and physical characteristics of ice cream cones. The 9% nanocalcium flour treatment was identified as the best formulation based on moisture content, calcium content, hardness, and resistance to ice cream, with values of $2.46 \pm 0.32\%$, $0.85 \pm 0.07\%$, 2081.63 ± 76.72 gf, and 47.33 ± 1.53 min, respectively. The hedonic acceptance of the 9% treatment ranged from $3.64 < \mu < 3.88$, with scores of 3.73 ± 0.58 for appearance, 3.80 ± 0.61 for aroma, 3.67 ± 0.48 for taste, and 3.83 ± 0.53 for texture. These findings indicate that blue swimming crab shell nanocalcium flour can

be used as a functional fortificant to improve the calcium content and physical performance of ice cream cones.

KEYWORDS: Blue swimming crab shell; nanocalcium flour; ice cream cone; calcium; physical characteristics.

1. INTRODUCTION

Blue swimming crab (*Portunus pelagicus*) is one of the fishery commodities widely consumed by the public, particularly as peeled crab meat in canned products. Based on data from the Ministry of Marine Affairs and Fisheries, the export value of blue swimming crab increased by an average of 7.38% during 2010-2015 [1]. As blue swimming crab is commonly exported in processed forms, particularly canned crab products, the increasing export value is expected to generate a corresponding increase in crab shell by-products.

Blue swimming crab shell flour is a potential alternative for utilizing shell waste generated by crab mini-plants. The incorporation of crab shell flour into food products can improve nutritional value, particularly calcium content. Crab shells contain minerals primarily in the form of calcium carbonate and, to a lesser extent, calcium phosphate. Calcium analysis on a dry-weight basis has shown that flour from internal crab waste contains 14.87% calcium, whereas shell waste flour contains 39.32% calcium [2].

Calcium is an essential macromineral required by the human body in amounts greater than 100 mg per day. The recommended calcium intake for the Indonesian population is 1000-1200 mg/day [3]. Calcium plays a fundamental role in the formation and maintenance of bones and teeth, and inadequate intake may therefore have implications for skeletal health.

Ice cream is one of the most popular food products in Indonesia. Gelato served in an edible cone has also become increasingly popular among consumers. The addition of blue swimming crab shell flour in nano-sized form may offer a practical approach to enhancing the nutritional value of ice cream cones. Previous research has indicated that smaller powder particle size can improve calcium absorption, and nanocalcium has been reported to enhance calcium bioavailability [4].

The addition of blue swimming crab shell flour is expected to increase the nutritional quality of ice cream cones, particularly their calcium content, while also influencing the physical characteristics of the cone matrix. Previous work reported significant differences in the calcium content of ice cream cones fortified with 5% shrimp shell flour, while cones fortified with 10% shrimp shell flour remained acceptable to panelists [5]. Therefore, this study was

conducted to evaluate the effect of blue swimming crab shell nanocalcium flour addition on calcium content and physical characteristics of ice cream cones.

2. MATERIALS AND METHODS

2.1. Materials

The materials used in this study included blue swimming crab shells obtained from a mini-plant in Demak, wheat flour, egg white, powdered sugar, butter, vanilla essence, and baking soda. All materials used in cone preparation were food-grade materials, while chemicals used for analytical procedures were prepared according to the respective methods described below.

2.2. Preparation of Blue Swimming Crab Shell Nanocalcium Flour

Nanocalcium flour was prepared using a method adapted from Lekahena et al. [6], with modification in the soaking process. Crab shells were first cleaned, washed, and boiled at 100°C for 30 min. The shells were then reduced in size and soaked in 1 N HCl solution at a sample-to-solvent ratio of 1:3 for 48 h for extraction. The extracted shells were cooled, filtered, and neutralized using distilled water until neutral pH was achieved. The material was then dried in an oven at 50°C for 12 h and milled using a ball mill to produce nanocalcium flour.

2.3. Preparation of Ice Cream Cones

The ice cream cone formulation was based on Utama [7]. Blue swimming crab shell nanocalcium flour produced in the previous stage was added to the cone dough at concentrations of 3%, 6%, and 9% relative to the amount of wheat flour used in the formulation. Cone preparation followed the creaming method. In this method, sugar and egg are first mixed until expanded, followed by mixing margarine, butter, and baking powder until a pale color is achieved; dry ingredients and other components are subsequently added and mixed until homogeneous [8].

Approximately one spoonful of dough (50 mL) was poured into the cone maker after the indicator light changed from green to red. Baking was conducted for 2 min, after which the sheet was removed while still hot. Because the cone structure remained pliable immediately after baking, it was shaped into a cone by rolling it using a cone roll.

2.4. Particle Size Analysis

Particle size analysis was conducted using a Zetasizer Nano based on the Malvern method [9]. The sample was dispersed in a liquid medium prior to measurement. The prepared sample was then placed into the instrument, and measurement was initiated using the software command. The resulting particle size data were displayed on the computer monitor.

2.5. Moisture Content Analysis

Moisture content was determined according to AOAC [10] by evaporating the water contained in the sample. The empty dish was dried in an oven at 100-105°C for 30 min, cooled in a desiccator for 30 min, and weighed (A). Approximately 2 g of sample was placed in the dried dish and weighed (B), then dried in an oven at 100-105°C until constant weight was reached (8-12 h). The sample was cooled in a desiccator for 30 min and weighed again (C). Moisture content was calculated using the following equation:

$$\text{Moisture content (\%)} = [(B - C) / (B - A)] \times 100$$

where A is the weight of the empty dish, B is the weight of the dish plus initial sample, and C is the weight of the dish plus dried sample.

2.6. Calcium Content Analysis

Calcium content was analyzed according to Apriyantono et al. [11]. A 5 g sample was ashed and then destructed by adding HNO₃:H₂O (10:30) for 10 min. The stock solution was prepared by cooling and filtering the destructed sample into a 50 mL volumetric flask, followed by dilution with demineralized water to the calibration mark. The stock solution was diluted by transferring 0.5 mL into a 10 mL volumetric flask and adding demineralized water to the calibration mark. For measurement, 0.5 mL of the diluted solution was transferred into a 10 mL volumetric flask, followed by the addition of 2 mL lanthanum and demineralized water to the calibration mark. Calcium was measured using an atomic absorption spectrophotometer (AAS) at a wavelength of 422.7 nm. The calcium concentration obtained from AAS was calculated using the following equation:

$$\%Ca = (\text{Concentration} \times \text{stock solution volume} \times \text{dilution factor}) / (\text{Sample weight} \times 10,000)$$

2.7. Hardness Analysis

Hardness was measured using the method of Saeleaw and Schleining [12]. Force applied to the product compressed the structure until its critical point was reached, causing fracture. Hardness testing was conducted using a TA-XT texture analyzer equipped with a spherical ball probe. The sample was placed on an aluminum plate with an opening that allowed the probe to penetrate the sample after compression.

2.8. Resistance to Ice Cream

Resistance of the cone to ice cream was evaluated using the method of Prihastuti [13]. The cone was filled with ice cream, and the time required until the cone became leaky and softened, thereby losing its ability to support the ice cream, was recorded.

2.9. Hedonic Evaluation

Hedonic evaluation was conducted according to Negara et al. [14]. Sensory responses were analyzed using a score sheet with a five-point preference scale: 1 = strongly dislike, 2 = dislike, 3 = neutral, 4 = like, and 5 = strongly like.

2.10. Statistical Analysis

Parametric data, consisting of moisture content, calcium content, hardness, and resistance to ice cream, were analyzed using analysis of variance (ANOVA). When significant differences were found, further analysis was conducted using the HSD test. Non-parametric hedonic data were analyzed using the Kruskal-Wallis test, followed by the Mann-Whitney test when significant differences were detected. Statistical significance was set at $p < 0.05$.

3. RESULTS AND DISCUSSION

3.1. Characteristics of Blue Swimming Crab Shell Nanocalcium Flour

The characteristics of blue swimming crab shell nanocalcium flour are presented in Table 1.

Table 1. Characteristics of blue swimming crab shell nanocalcium flour.

No.	Parameter	Result
1.	Yield	41.33%
2.	Particle size	488.2 nm
3.	Calcium content	19.41%

Blue swimming crab shell nanocalcium flour had a yield of 41.33%. Soaking in 1 N HCl for 48 h was presumed to influence the resulting yield. Previous research on shrimp shell nanocalcium showed that 48 h soaking produced the most optimal yield compared with 24 h and 72 h soaking [15]. Yield may increase until concentration equilibrium or saturation is reached in the solution, and solvent concentration may also affect the yield by influencing the solubility of shell components [16].

The particle size of blue swimming crab shell nanocalcium flour was 488.2 nm, which falls within the nanoscale range because it is below 1000 nm. Nanoparticles are generally defined as particles with sizes ranging from 10 to 1000 nm [17]. One factor influencing the particle size of the nanocalcium flour was the use of ball milling. The ball mill method is advantageous in flour production because it can produce finer particles [18].

The calcium content of the nanocalcium flour was 19.41%, equivalent to 19,419 mg/100 g. The calcium content of crab shell flour may be influenced by the solvent used during flour preparation. The use of HCl in nanocalcium preparation has been reported to increase both yield and calcium value [15].

3.2. Moisture Content

The moisture content of ice cream cones ranged from 6.40% to 2.46%. Moisture content decreased as the concentration of blue swimming crab shell nanocalcium flour increased.

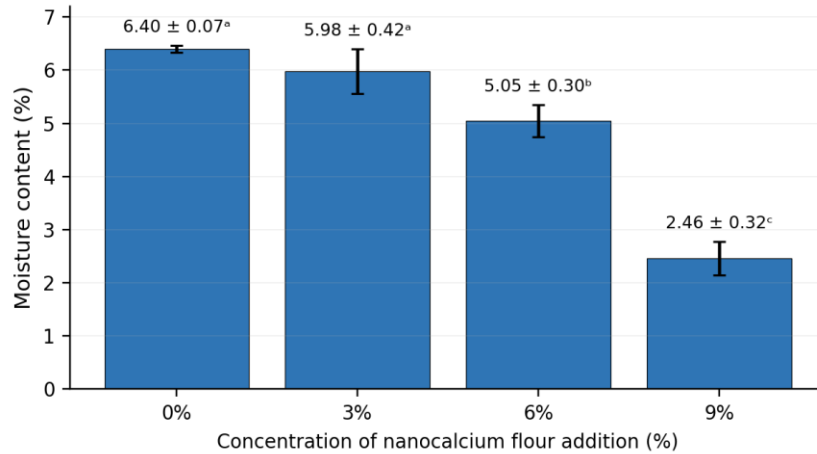


Figure 1. Moisture content of ice cream cones fortified with blue swimming crab shell nanocalcium flour.

The decrease in moisture content in ice cream cones fortified with blue swimming crab shell nanocalcium flour may be attributed to the increasing amount of solid material in the cone dough. The addition of pokea shell flour has similarly been reported to reduce moisture content compared with the control treatment without shell flour addition [19].

The reduction in moisture content was also associated with the calcium contained in the nanocalcium flour. The addition of fish bone flour containing calcium may increase Ca^{++} particles in the product, which subsequently bind OH^- particles as part of water molecules, thereby reducing moisture content [20].

3.3. Calcium Content

The lowest calcium content in ice cream cones was $0.02 \pm 0.01\%$, equivalent to 20 mg/100 g. The highest calcium content was observed in the 9% nanocalcium flour treatment, with a value of $0.85 \pm 0.07\%$, equivalent to 850 mg/100 g. Considering that the recommended calcium intake for Indonesians is 1000-1200 mg/day [3], the addition of crab shell flour to ice cream cones may contribute meaningfully to dietary calcium intake.

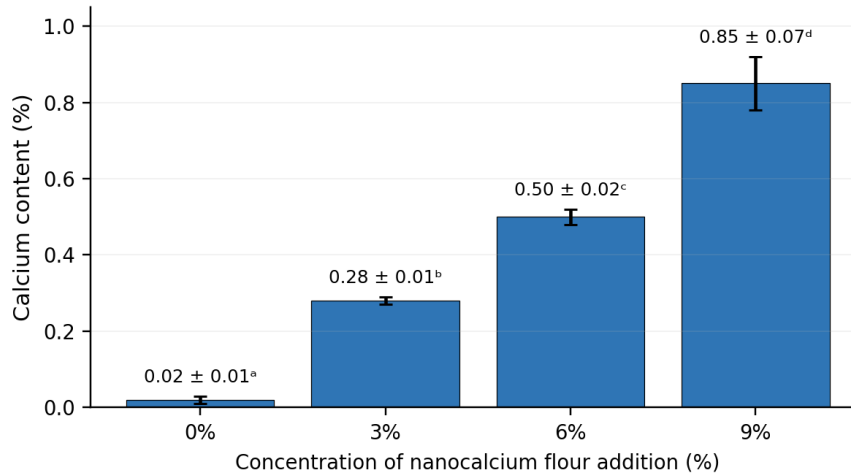


Figure 2. Calcium content of ice cream cones fortified with blue swimming crab shell nanocalcium flour.

Calcium content in ice cream cones increased with increasing concentrations of blue swimming crab shell nanocalcium flour. A similar tendency has been reported in cookies fortified with milkfish bone flour, where calcium content increased as the concentration of bone flour increased [21]. This increase was caused by the incorporation of a calcium-rich material into the formulation. Control cones without nanocalcium flour also contained a small amount of calcium, which may have originated from butter, a dairy-derived ingredient. The addition of ingredients such as milk and butter can influence calcium content because dairy products contain relatively high levels of calcium [22].

Calcium absorption is strongly affected by phosphorus and vitamin D status in the body. Calcium deficiency may contribute to osteoporosis, whereas excessive calcium intake may cause hypercalcemia and kidney disorders [23]. Vitamin D enhances intestinal mucosal absorption by stimulating the production of calcium-binding proteins. A calcium-to-phosphorus intake ratio of 1:1 is recommended to support optimal utilization [24].

3.4. Hardness

Hardness testing showed that ice cream cones without blue swimming crab shell nanocalcium flour had the lowest hardness value, whereas cones with 9% nanocalcium flour had the highest hardness value.

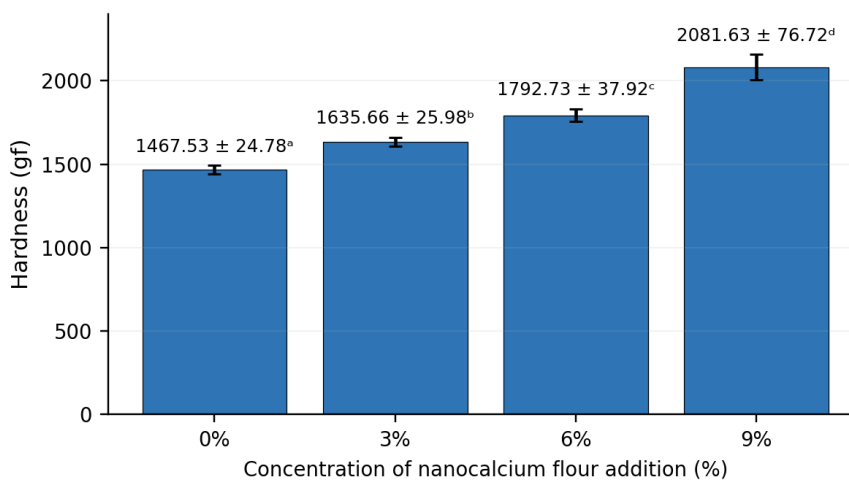


Figure 3. Hardness of ice cream cones fortified with blue swimming crab shell nanocalcium flour.

The hardness values obtained in this study indicate that increasing nanocalcium flour concentration increased cone hardness, primarily due to the relatively high calcium content of the flour. The presence of calcium in tilapia bone flour has been reported to increase the calcium content of wonton crackers, and higher calcium content was associated with a harder texture [25].

In addition to calcium addition, cone hardness was also influenced by the high temperature applied during baking. High baking temperature evaporates water from the cone dough, and greater water loss results in a harder cone. Baking temperature and time can also affect biscuit hardness because rapid heating at high temperatures causes substantial textural changes; low moisture content generally produces a harder texture [26,27].

The control cone had a hardness value of 1467.53 gf. This value was associated with the use of wheat flour, which functions as the structural framework of the cone. Wheat flour contains gluten-forming proteins, namely gliadin and glutenin, which form gluten upon hydration and mixing. When heated, gluten contributes to hardening of the product structure [28].

3.5. Resistance to Ice Cream

The ability of the cone to support ice cream ranged from approximately 23 to 47 min.

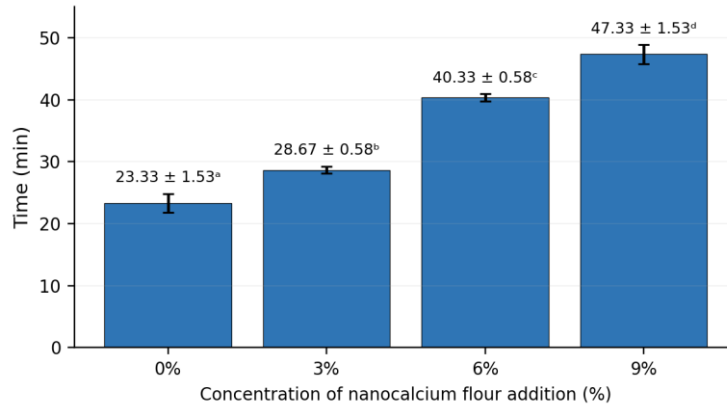


Figure 4. Resistance of ice cream cones to ice cream.

The ability of the cone to support ice cream was determined by cone hardness and water absorption capacity, or hygroscopicity. Cone hygroscopicity is influenced by environmental conditions and by melted ice cream. Melted ice cream causes the cone to soften because the cone absorbs water from the ice cream. Water absorption capacity is particularly influenced by the environment in low-moisture products, as the product absorbs moisture to reach equilibrium, resulting in loss of crispness [27].

Water absorption capacity in ice cream cones may also be affected by protein content. Increasing concentrations of fish flour in ice cream cone production have been reported to prolong the ability of the cone to support ice cream, presumably due to the presence of hydrophobic proteins [29]. The addition of 7% blue swimming crab shell flour has also been reported to increase protein content in cheese sticks to 12.29% [30].

3.6. Hedonic Evaluation

Hedonic testing was conducted with 30 panelists. Each panelist assessed the appearance, aroma, taste, and texture of the tested cones.

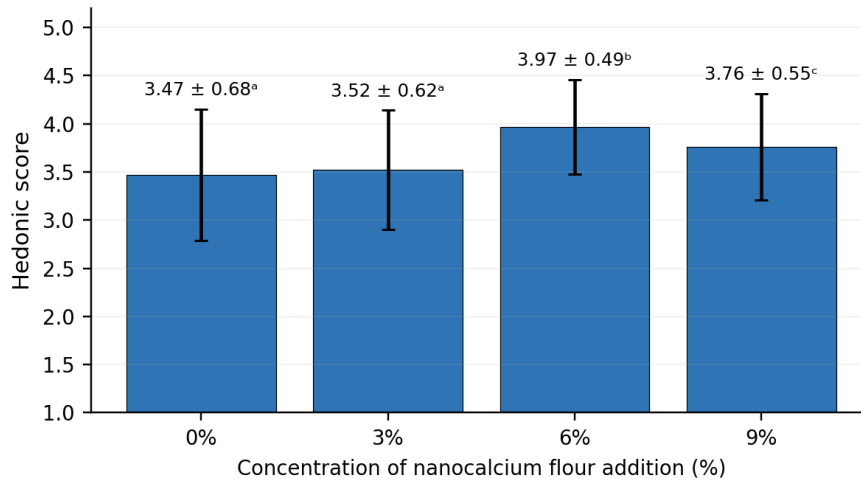


Figure 5. Hedonic scores of ice cream cones fortified with blue swimming crab shell nanocalcium flour.

The data indicate that ice cream cones fortified with different concentrations of blue swimming crab shell nanocalcium flour were acceptable to panelists. This was reflected in mean hedonic scores ranging from 3.47 to 3.97 across treatments. Significant differences were observed in the 6% and 9% treatments, whereas the control and 3% treatment did not differ significantly. The highest hedonic score was obtained in the 6% nanocalcium flour treatment, with a value of 3.97 ± 0.49 .

3.6.1. Appearance

Panelists evaluated cone appearance based on color, uprightness, and physical integrity.

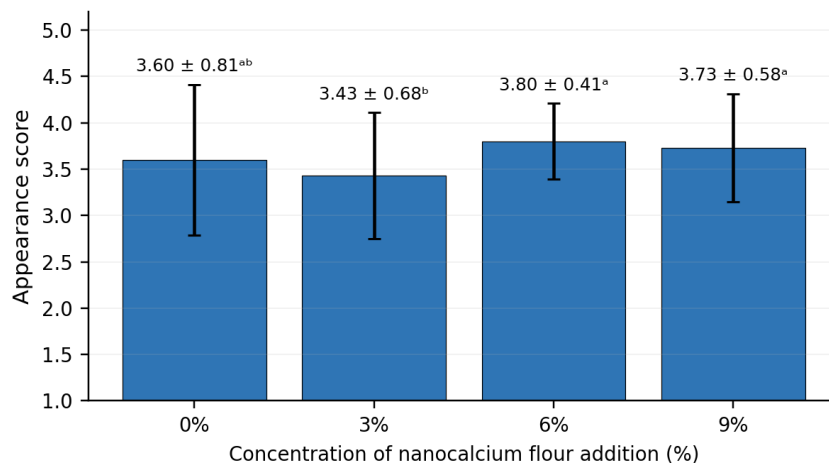


Figure 6. Hedonic scores for the appearance of ice cream cones.

Ice cream cones with 3% nanocalcium flour differed significantly from cones with 6% and 9% nanocalcium flour. This difference may be related to increased protein content, which promoted browning through the Maillard reaction during baking. Substitution with catfish meat flour has been reported to increase protein content in biscuits, and high-temperature baking can produce a brown color through reactions between carbohydrates and primary amino groups [31].

3.6.2. Aroma

Panelists assessed aroma based on the odor characteristics of the ice cream cones.

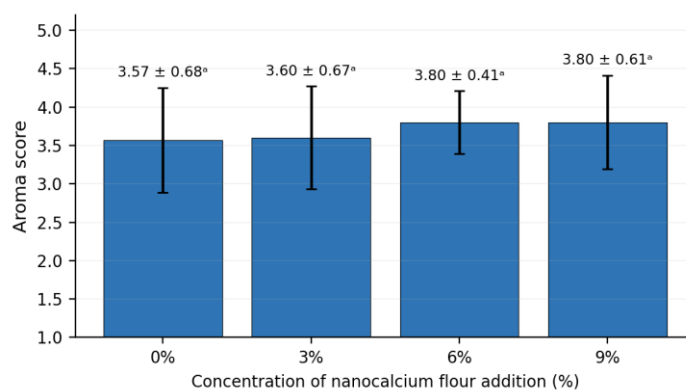


Figure 7. Hedonic scores for the aroma of ice cream cones.

The hedonic aroma results showed that the aroma of cones from all treatments was acceptable to panelists. No significant differences were observed among treatments because the concentrations of nanocalcium flour added were relatively low. Cone aroma was more strongly influenced by ingredients such as butter and vanilla essence. Butter, as a dairy derivative, contains volatile compounds including lactic acid, diacetyl, and lactose; these volatile compounds evaporate and contribute to biscuit aroma [32].

3.6.3. Taste

Taste evaluation was based on the perceived flavor of the ice cream cones.

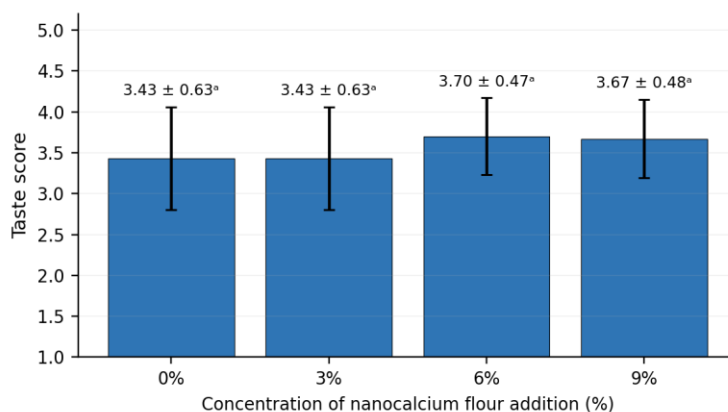


Figure 8. Hedonic scores for the taste of ice cream cones.

The results showed no significant differences in taste among treatments. Nanocalcium flour addition did not markedly affect cone taste because powdered sugar was added at a higher proportion than the nanocalcium flour. Consequently, the resulting cones were dominated by sweetness. Flour used in biscuit preparation generally has a bland taste, and the addition of powdered sugar across all formulations contributes sweetness without generating significant differences in flavor [33].

3.6.4. Texture

Panelists assessed texture based on the hardness and mouthfeel of the ice cream cones.

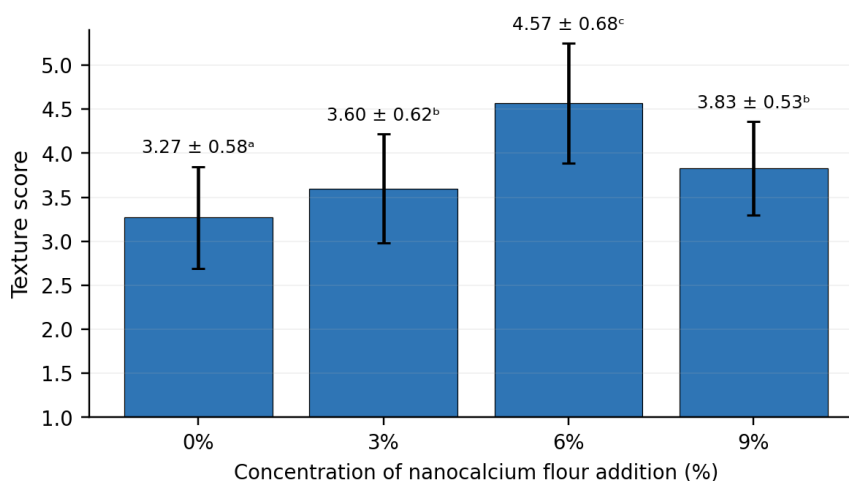


Figure 9. Hedonic scores for the texture of ice cream cones.

The hedonic texture data showed that panelists accepted all cone treatments. The highest texture preference score was obtained in cones fortified with 6% nanocalcium flour, with a value of 4.57 ± 0.68 , whereas the lowest score was observed in the control treatment, with a value of 3.27 ± 0.58 . This indicates that panelists preferred cones with a harder texture

produced by nanocalcium flour addition. Product texture is influenced by ingredient composition and heating processes, and is strongly affected by moisture content, fat content, and the types of carbohydrate and protein present in the formulation [34].

4. CONCLUSIONS

The addition of blue swimming crab shell nanocalcium flour affected the calcium content and physical characteristics of ice cream cones. The best treatment was obtained with 9% nanocalcium flour addition, which produced moisture content, calcium content, hardness, and resistance to ice cream values of $2.46 \pm 0.32\%$, $0.85 \pm 0.07\%$, 2081.63 ± 76.72 gf, and 47.33 ± 1.53 min, respectively. The hedonic acceptance of ice cream cones with 9% blue swimming crab shell nanocalcium flour ranged from $3.64 < \mu < 3.88$. These results indicate that blue swimming crab shell nanocalcium flour can serve as a functional calcium fortificant for ice cream cones while improving selected physical properties of the product.

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