Development and characterization of Ice cream using Eggshell Powder to Curtail Calcium Deficiency

1. **Ali Hasan**
alihasanfst@gmail.com
2. **Yasmeen Bano**
yasmeen@uaf.edu.pk
3. **Saira Kazim**
sairakazim193@gmail.com
4. **Muhammad Usman**
m.usmanshafiq870@gmail.com
5. **Tanveer Ahmad**
tanveerahmad7304@gmail.com
6. **Rizwan Shukat***
RIZWANUAF@HOTMAIL.COM
7. **Syed Qamar Abbas**
Qamar.Abbas@uaf.edu.pk
8. **Muhammad Ali**
muhammadali.uvas@gmail.com

1,3,4,5,6,7) National Institute of Food Science and Technology, University of Agriculture Faisalabad
2) Department of Food Science and Technology, University of Agriculture Faisalabad Sub-Campus Burewala
8) Department of Dairy Technology, University of Veterinary and Animal Sciences Lahore

*Correspondence Author
RIZWANUAF@HOTMAIL.COM

Abstract

Calcium plays an important role in bone and teeth formation. Its deficiency can lead to severe bone and metabolic disease. There is a variety of calcium sources available and most of them are expensive. The eggshell contains 94% calcium carbonate in it and is suitable for human consumption. It is the most economical calcium source and can be added to ice cream. Ice cream, a complex frozen food colloid, is the most popular dessert in the world. Food technologists have done several studies on it using it as a functional food.The objective of this study was to evaluate the effect of eggshell powder as a source of calcium and to check out the stability of ice cream. Eggshell powder was added in ice cream as a calcium source. Skim milk powder was replaced with eggshell powder with 10%, 20%, 30%, and 40% concentrations for T_1, T_2, T_3, and T_4 respectively.Proximate analysis, textural analysis, physiochemical

analysis including overrun, viscosity, melting rate, and sensory analysis were done to check the stability of ice cream. The results showed that the increase of eggshell powder enhanced the mineral density of ice cream and increased calcium from 440mg/100g in T_0 to 740mg/100g in T_4. There was a decrease in protein, fat, TSS, pH, Overrun, and hardness. The results also conclude an increase in ash, titratable acidity, viscosity, melting rate. The sensory panel also revealed that the addition of eggshell is beneficial for ice cream characteristics. Among the ice cream samples, T_1 showed more acceptability. It is recommended that eggshell powder should be replaced up to 10% with skim milk in ice cream preparation

Key Words: **Ice cream, eggshell, calcium**
DOI Number: 10.48047/nq.2023.21.6.NQ23030 **NeuroQuantology2023;21(6):258-274**

Introduction

Ice cream is a famous dairy product and its history is centuries old. Ice cream was first served some 3000 years ago to the emperor of China. Chinese people mixed fruit juices with ice and considered it as frozen food. According to roman historical writers, centuries-old traditions involved the cooling of food and beverages with snow which resulted in the production of ice cream. In thirteen centuries, Marco polo came back to Italy with the recipes of water ices that has been used by Asian peoples for thousands of years. New processes for freezing of water with the help of salt and ice revolutionized the modern ice cream manufacturing (Goff and Hartel, 2013).

The science behind ice cream is very simple, from the ingredients in ice cream, fat globules give creamy mouthfeel related to ice cream. Air is inoculated so that air makes up 50% of volume and gives ice cream texture. Casein and whey proteins on the fat globules keeps them well distributed. Sugar sweetens the ice cream and lowers the freezing point. Lots of small ice crystals makes smooth texture. This happens as a result of the speed of freezing. Fast freezing makes small crystals.The preparation of ice cream consists of two major processes. The first process proportion of different ingredients is calculated to prepare the formulation mix. All ingredients are multifunctional, provide quality to the product, and are helpful in the stability of ice cream. In the second step, all the ingredients are mixed according to the calculated recipe.

Global egg consumption and production have increased dramatically since the turn of the century. In 2018, global egg production reached 76.7 million tones, a rise of 14.95 percent over the previous ten years. For the past thirty years, China has been the world's greatest producer of eggs, with the total output hitting a peak of 31 million tons in 2016. China was responsible for 26.90 million tons of output in 2018. In the same year, the United States of America (USA) came in second with a gross yearly output of 6.46 million tones, followed by India with a gross annual production of 5.23 million tones, Mexico with 2.87 million metric tons, Brazil with 2.66 million tons and Japan with 2.62 million tons.Eggshell makes up around 10% of the weight of a hen's egg. Large quantities of chicken eggs are produced each year, with a major fraction (30%) processed in food processing plants, resulting in a vast buildup of eggshell trash(Aditya *et al.*, 2021).

Calcium is the most prevalent mineral in the human body. It eventually contributes to bone production, contributing for 99 percent of all bones and teeth. A little percentage of calcium (1%) is located outside of the skeletal tissue, which serves a variety of purposes. Calcium obtained from dairy sources is sufficient to meet the body's calcium needs. However, individuals do not consume enough calcium as recommended by professional standards, resulting in severe bone thinning.Due to calcium shortage in postmenopausal women and osteoporosis patients, dietary calcium supplementation has increased. Furthermore, many individuals in Asia and Africa suffer from

lactose intolerance, necessitating the development of novel calcium supplements to meet dietary calcium requirements in the form of fortified dietary items.

Calcium is an important mineral associated with healthy bones and teeth. It is found in many foods, such as dairy products. It plays an important role in blood clotting, muscles contraction, and nerve functions. It is the most abundant mineral in the human body for playing a significant role in bone formation, 99% for teeth and bones. The daily average intake is between 1000 and 1300 mg depending upon age and sex(Costa *et al.*, 2008). Most people, who do not meet calcium requirements, are at the stage of bone mass development, especially in underdeveloped countries. Fortification is necessary to meet calcium requirements to reduce calcium deficiency, and avoid diseases like osteoporosis. Calcium helps in maintaining metabolic processes like blood clotting, cell adhesion, muscle contraction, hormone and neurotransmitter release, glycogen metabolism, and cell proliferation.

The bioavailability of calcium from ice cream equals that of milk despite the effects of higher fat on reduced calcium solubility. In milk, ⅓ of calcium in milk is soluble and the remaining ⅔ plays an important role in micelle structure and stability, as it is associated with casein micelles as colloidal calcium phosphate or calcium ions bound to phosphoserine residues (⅙ of total calcium) (Costa *et al.*, 2008). After knowing the value of calcium available in eggshell and desirability of ice cream by every age group, this study has been planned to curtail calcium deficiency especially osteoporosis. The need of the project is that mostly people do not meet their RDA of calcium. Mostly calcium sources available are expensive and utilization of eggshell is an economical way to meet calcium RDA.

Objectives of the study are given below.

- To develop calcium-fortified ice cream by adding various concentrations of eggshell powder
- To evaluate the stability of ice cream during storage
- To evaluate the effect of eggshell powder as a calcium source on physicochemical and sensory characters of Ice cream.

Methodology

Procurement of raw materials

Skim milk, cream, sugar, and stabilizer were purchased from the chase up store in Faisalabad and brought to the dairy technology laboratory for research. Eggshells were procured from the canteen in the university.

Preparation of eggshell powder

Eggshells were washed properly to wipe out the dirt on them. After that, the inner shell membrane was separated and eggshells were boiled for 30-35 minutes to kill physical contaminants. After that, eggshells were placed to dry. When the egg shells were completely dried, they were sterilized in an autoclave at 121°C for 30 minutes. When the eggshells were dried, they were kept in hot air oven at 105°C for 2hrs to remove moisture completely. When they were dried completely, eggshells were ground by using a kitchen grinder. They were passed from a sieve to get smooth and fine particles. The fine particles were stored in plastic bags.

Ice formation process

Recipe of ice cream formation and different levels of eggshell concentrations replacement with skim milk powder is given in the Table (a) and (b). For ice cream formation skim milk was replaced with 4 different concentrations of egg shells.

Table: (a) Ice cream formation

Ingredients	**Percentage**

Water	62.5
Skim milk powder (SNF)	12
Fat	12
Sucrose	13
Stabilizer	0.5

Treatment plan

Treatment	**Skim milk powder (SNF)%**	**Eggshells%**
T_0	100	0
T_1	90	10
T_2	80	20
T_3	70	30
T_4	60	40

Flow diagram for the ice cream preparation

Flow process for ice cream preparation is illustrated below.

Ingredients Mixing

Pasteurization 70°C

Homogenization

Cooling 4°C

Ice cream mix + Eggshell powder

Aging 24hrs

Freezing and Storage -18°C(Ismail *et al.*, 2020)

Figure (a)

Physiochemical analysis of ice cream

Moisture content

The moisture content of ice cream was assessed by AOAC (2016). At first, the sample was weighed and set in the hot air oven in an oven-dried China dish at the temperature of 105°Cfor 2 hours. After the length of 24 hours, the dried China dish was removed from a hot air oven and put in the desiccator to avoid moist.

$$\text{Moisture\%} = \frac{\text{Weight of original sample(g)} - \text{Weight of dried sample (g)}}{\text{Weight of original sample (g)}} \times 100$$

Ash Content

Ash content of ice cream was determined by using standard given by AOAC (2016). The sample of ice cream (1g) was placed in a pre-weighed crucible and burnt on Bunsen burner for 10-15 minutes until the exhaust emerging from the cauldron vanished. In the wake of the scorching and charring, the sample of kabab was put in Muffle Furnace for going through direct burning at 550-600°C till white grisly, gloomy and miserable slag was left. The crucible was weighed in the wake of cooling in desiccators. Ash was determined by the equation composed underneath

$$\text{Ash\%} = \frac{\text{Weight of ash in sample (g)}}{\text{Weight of original sample (g)}} \times 100$$

Crude Fat

Fat in the ice cream was determined by the Gerber method followed by AOAC (2016). For the determination of fat, the 10 ml H_2SO_4 solution was measured carefully with the help of pipette and poured into the butyrometer. Then add 1ml of Isoamyl alcohol and 10.94 ml of milk sample. When the stopper adjust carefully, butyrometer was shake for the mixing of the sample. Then sample was placed in the Gerber machine. The Gerber machine was set on 65°C for 5 minutes at 1100 rpm. Remove the butyrometer from Gerber machine. Fat layer was separated. The fat layer was noted in butyrometer scale carefully against graduation scale.

Crude Protein

The crude protein of eggshell fortified ice cream was determined by method described by AOAC (2016). First of all, a clean digestion tube was taken and one digestion tablet, three grams of sample, and 30ml of H2SO4 were added into it. Slow heated the sample for 45 minutes to start the digestion process and to prevent bubbles formation and then it was heated until the pale green color was observed. After the digestion was completed, the digestion sample was placed to cool down. A volumetric flask of 250ml volume was taken, washed 2-3 times with distilled water and the digested sample was shifted to the flask. Distilled water was incorporated into the flask to raise the volume of the mixture to 250ml. then 10ml of NaOH along with the 10ml of sample from the digested mixture was added to the distillation unit. On the other side, a beaker containing a 4% boric acid solution and few drops of methyl red as an indicator was placed in which ammonia produced in the distillation unit was collected. Turning of red to yellow was the end point of the reaction and this process. The last process was titration of distilled content against 0.1 N sulfuric acid with pink color as the endpoint. The volume used of sulfuric acid was noted.

First of all, total nitrogen content was calculated using the following formula and then its value

was multiplied with a factor to determine total protein.

$$\text{Nitrogen } \% = \frac{\text{H2SO4(ml)} \times 250 \times 0.0014}{\text{Weight sample } \times \text{sample volume (digested)}} \times 100$$

$$\text{Total protein } \% = \text{Nitrogen}\% \times 6.38$$

pH analysis

A digital pH meter was used to analyze the pH of samples. First of all, two buffer solutions of pH 4.0 and 7.0 were taken to standardize the equipment. Samples were taken in a glass beaker and the temperature adjusted at 25°C. The electrode of the pH meter was put in the ice cream sample and the digital value was noted until it became constant. (Han *et al.,* 2006)

Titratable acidity

The titration method was used to calculate the acidity of ice cream by adopting the procedure describe by AOAC (2016). Samples of 10 ml into the beaker proceeded by adding 2-3 drops of phenolphthalein indicator and titrated against 0.1 N of NaOH unless pink color occurred. The acidity was determined by the formula as following:

$$\text{Acidity } \% = \frac{0.009 \ \times \text{vo. of 0.1 N NaOH used}}{\text{Sample Weight}} \times 100$$

Total soluble solids

Total soluble solids in the ice cream were measured by using refractometer. The value was calculated in the unit brix. The procedure was followed according to Halim *et al.,* (2014).

Overrun

An increase in the volume of ice cream achieved during the process of freezing owing to the inclusion of air is called overrun. The overrun of ice cream was determined by the method of Sofjan and Hartel (2004) using the below mentioned formula.

Textural analysis of ice cream

$$\text{Overrun } \% = \frac{\text{Weight of mix} - \text{eggshell added ice cream}}{\text{Weight of mix}} \times 100$$

Viscosity

Ice cream viscosity was measured using viscometer Brookfield LVDVE (MA, USA). Containers of 180ml volume and 6 cm diameter × 9cm height filled with ice cream was taken at 6°C ± 1°C. The viscometer was operated at 20 revolutions per minute with spindle number 4(Sofjan and Hartel, 2004).

Hardness

The textural analyzer (TA-XT2i, Stable Microsystems Ltd., UK) was used to determine the hardness of the ice cream. The ice cream was added to a 33ml cylindrical container and readings were taken at a temperature of 25^0C ± 2^0C with the help of a textural analyzer, equipped with a 5mm, test speed 3mm/s, posttest speed 10mm/s, data acquisition rate 250 pps and trigger type auto 20 g (Hanafi *et al.*, 2022).

Melting rate

The meltdown time of ice cream was evaluated by the technique described by Clark (2015) up to 60 minutes. About 25 grams of ice cream was taken on 2mm sieves and a beaker was kept under the sieve to receive melting drops of ice cream at a constant temperature of 25°C. Using a stopwatch time of 60 minutes was calculated and the volume of ice cream that was melted down is measured by the volumetric cylinder. To get an average value process was repeated for three times. Standup time is that when the first drop of ice cream fell in beaker.

Mineral analysis

Mineral analysis for the determination of calcium content was measured by using flame photometer according to the method described by AOAC (2016). Before passing the sample from flame photometer, digestion of the sample was done. For this 0.5g of sample was digested using per chloric acid and nitric acid. After this the filtrate was added in the distilled

water and dilution was done. After that, calcium content was measured.

Sensory evaluation

Sensory evaluation of different parameters including appearance, texture, mouthfeel, flavor, and overall acceptability was conducted using the 9-point hedonic scale technique defined by Civille and Oftedal (2012).

Results

Chemical composition of skim milk powder, cream, and eggshell

Constituent	Skim milk powder	Cream	Eggshell powder
Total solids (%)	96.5 ± 0.5	35 ± 0.5	97.5±0.5
MSNF (%)	96 ± 0.4	5 ± 0.6	-n.d-
Moisture (%)	3.5 ± 0.5	65 ± 0.5	2.5±0.5
Ash (%)	8.4 ± 0.5	0.03 ± 0.02	93.5±0.5
Protein (%)	31.8 ± 0.08	2.2 ± 0.1	3.5 ± 0.5
Fat (%)	0.5 ± 0.2	30 ± 0.5	1.0 ±0.2
Calcium	0.8 ± 0.2	-n.d-	88±3.5

*MSNF= Milk solids not fat *n.d= Not determined

Mean values for the protein

Treatment	Days				Mean
	0	7	14	21	
T_0	4.3 ± 0.05	4.27 ± 0.03	4.35 ± 0.07	4.23 ± 0.06	4.29 ± 0.05^{a}
T_1	4.1 ± 0.06	4.05 ± 0.06	4.09 ± 0.07	4.04 ± 0.07	4.07 ± 0.02^{b}
T_2	3.79 ± 0.06	3.72 ± 0.09	3.67 ± 0.13	3.76 ± 0.07	3.73 ± 0.05^{c}
T_3	3.52 ± 0.09	3.45 ± 0.05	3.81 ± 0.7	3.33 ± 0.08	3.53 ± 0.2^{d}
T_4	3.32 ± 0.08	3.31 ± 0.09	3.22 ± 0.09	3.18 ± 0.07	3.26 ± 0.07^{e}
Mean	3.81 ± 0.4^{a}	3.76 ± 0.39^{a}	3.83 ± 0.42^{a}	3.7 ± 0.44^{a}	

T_0 = Ice cream with 0% eggshell powder
T_1 = Ice cream with 10% of eggshell powder replaced with skim milk powder
T_2 = Ice cream with 20% of eggshell powder replaced with skim milk powder
T_3 = Ice cream with 30% of eggshell powder replaced with skim milk powder

T_4 = Ice cream with 40% of eggshell powder replaced with skim milk powder

Mean values for the crude fat

Treatment	**Days**				**Mean**
	0	**7**	**14**	**21**	
T_0	11.73 ± 0	11.72 ± 0	11.71 ± 0	11.72 ± 0	11.72 ± 0[a]
T_1	11.18 ± 0.02	11.18 ± 0.01	11.2 ± 0.01	11.18 ± 0.02	11.18 ± 0[b]
T_2	10.83 ± 0.02	10.84 ± 0.01	10.84 ± 0.01	10.83 ± 0	10.84 ± 0[c]
T_3	10.23 ± 0.01	10.23 ± 0	10.22 ± 0.01	10.24 ± 0.01	10.23 ± 0[d]
T_4	10.15 ± 0	10.14 ± 0	10.15 ± 0	10.15 ± 0	10.14 ± 0[e]
Mean	10.82 ± 0.66[a]	10.82 ± 0.66[a]	10.82 ± 0.66[a]	10.82 ± 0.65[a]	

Mean values for ash content

Treatment	**Days**				**Mean**
	0	**7**	**14**	**21**	
T_0	0.76 ± 0.01	0.77 ± 0.02	0.76 ± 0.02	0.79 ± 0.02	0.77 ± 0.01[a]
T_1	0.88 ± 0.01	0.89 ± 0.02	0.88 ± 0.01	0.88 ± 0.01	0.88 ± 0[b]
T_2	1.01 ± 0.01	1.03 ± 0.02	1.02 ± 0.01	1.03 ± 0.02	1.02 ± 0[c]
T_3	1.12 ± 0.02	1.13 ± 0.02	1.11 ± 0.01	1.13 ± 0.01	1.12 ± 0[d]
T_4	1.21 ± 0.02	1.19 ± 0.02	1.21 ± 0.02	1.19 ± 0.01	1.2 ± 0.01[e]
Mean	1 ± 0.18[a]	1 ± 0.17[a]	0.99 ± 0.17[a]	1 ± 0.16[a]	

Mean values for moisture content

Treatment	**Days**	**Mean**

	0	7	14	21	
T_0	64.5 ± 0.01	64.5 ± 0.01	64.5 ± 0.01	64.49 ± 0	64.49 ± 0[a]
T_1	64.34 ± 0	64.35 ± 0	64.34 ± 0	64.35 ± 0	64.34 ± 0[b]
T_2	64.2 ± 0	64.2 ± 0	64.19 ± 0	64.2 ± 0	64.2 ± 0[c]
T_3	64.1 ± 0	64.1 ± 0	64.09 ± 0	64.11 ± 0.01	64.1 ± 0[d]
T_4	63.95 ± 0	63.95 ± 0.01	63.95 ± 0.01	63.94 ± 0	63.94 ± 0[d]
Mean	64.22 ± 0.21[a]	64.22 ± 0.21[a]	64.21 ± 0.21[a]	64.22 ± 0.21[a]	

11 Mean values for TSS

Treatment	**Days**				**Mean**
	0	**7**	**14**	**21**	
T_0	31 ± 1	30.66 ± 0.57	30.33 ± 0.57	30 ± 0	30.5 ± 0.43[a]
T_1	29 ± 1	28.66 ± 0.57	28.33 ± 0.57	28.66 ± 0.57	28.66 ± 0.27[b]
T_2	26 ± 1	25.66 ± 0.57	25.33 ± 0.57	24.66 ± 0.57	25.41 ± 0.56[c]
T_3	25 ± 1	25.33 ± 0.57	25.16 ± 0.28	24.66 ± 0.57	25.04 ± 0.28[d]
T_4	24 ± 1	24.33 ± 0.57	23.83 ± 0.28	23.66 ± 0.57	23.95 ± 0.28[d]
Mean	27 ± 2.91a	26.93 ± 2.63[a]	26.6 ± 2.65[a]	26.33 ± 2.8[a]	

Mean values for pH

Treatment	**Days**				**Mean**
	0	**7**	**14**	**21**	

T_0	6.7 ± 0	6.69 ± 0.01	6.67 ± 0	6.65 ± 0	6.68 ± 0.02[a]
T_1	6.5 ± 0	6.49 ± 0	6.48 ± 0.01	6.47 ± 0	6.48 ± 0.01[b]
T_2	6.5 ± 0	6.49 ± 0	6.49 ± 0	6.48 ± 0	6.49 ± 0[b]
T_3	6.45 ± 0	6.44 ± 0.01	6.41 ± 0	6.39 ± 0	6.42 ± 0.02[c]
T_4	6.41 ± 0	6.41 ± 0.01	6.4 ± 0.01	6.39 ± 0	6.4 ± 0.01[d]
Mean	6.51 ± 0.11[a]	6.5 ± 0.1[a]	6.49 ± 0.11[b]	6.48 ± 0.1[c]	

Mean values analysis for titratable acidity

Treatment	**Days**				**Mean**
	0	**7**	**14**	**21**	
T_0	0.18 ± 0	0.18 ± 0	0.18 ± 0	0.18 ± 0	0.18 ± 0[a]
T_1	0.2 ± 0	0.2 ± 0	0.2 ± 0	0.2 ± 0	0.2 ± 0[b]
T_2	0.2 ± 0	0.2 ± 0	0.2 ± 0	0.2 ± 0	0.2 ± 0[c]
T_3	0.21 ± 0	0.21 ± 0	0.21 ± 0	0.21 ± 0	0.21 ± 0[d]
T_4	0.22 ± 0	0.22 ± 0	0.22 ± 0	0.22 ± 0	0.22 ± 0[e]
Mean	0.2 ± 0.01[a]	0.2 ± 0.01[ab]	0.2 ± 0.01[ab]	0.2 ± 0.01[b]	

Mean values table of melting rate

Treatment	**Days**				**Mean**
	0	**7**	**14**	**21**	
T_0	25.41 ± 0.38	25.25 ± 0.25	25 ± 0.25	24.75 ± 0.25	25.1 ± 0.29[e]
T_1	26 ± 0.25	25.75 ± 0.25	25.5 ± 0.25	25.25 ± 0.25	25.62 ± 0.32[d]

T_2	27.25 ± 0.25	27.16 ± 0.14	27 ± 0.25	26.75 ± 0.25	27.04 ± 0.22^{c}
T_3	27.91 ± 0.14	27.66 ± 0.14	27.33 ± 0.14	27 ± 0.25	27.47 ± 0.39^{b}
T_4	28.25 ± 0.25	28.16 ± 0.14	27.83 ± 0.14	27.66 ± 0.14	27.97 ± 0.27^{a}
Mean	26.96 ± 1.22^{a}	26.8 ± 1.25^{a}	26.53 ± 1.22^{b}	26.28 ± 1.23^{c}	

Mean values analysis of ice cream sample for viscosity

Treatment	**Days**				**Mean**
	0	**7**	**14**	**21**	
T_0	63.39 ± 0.34	65.56 ± 0.43	66.36 ± 0.55	66.84 ± 1	65.54 ± 1.52^{d}
T_1	67.33 ± 0.33	71.54 ± 0.43	72.5 ± 0.43	73.7 ± 0.27	71.27 ± 2.77^{b}
T_2	65.18 ± 0.16	67.81 ± 0.21	69.04 ± 0.34	69.91 ± 0.54	71.98 ± 2.06^{c}
T_3	68.03 ± 0.27	73.55 ± 0.38	75.7 ± 0.28	76.66 ± 0.3	73.48 ± 3.85^{a}
T_4	68.3 ± 0.16	74.39 ± 0.52	76.09 ± 0.29	77.19 ± 0.7	73.99 ± 3.96^{a}
Mean	66.44 ± 2.1^{d}	70.57 ± 3.77^{c}	71.94 ± 4.21^{b}	72.86 ± 4.43^{a}	

Mean values for the overrun of ice cream

Treatment	**Days**				**Mean**
	0	**7**	**14**	**21**	
T_0	52.13 ± 0.17	52.04 ± 0.06	51.97 ± 0	51.49 ± 0.03	51.9 ± 0.28^{a}
T_1	50.23 ± 0.22	50.04 ± 0.09	49.84 ± 0.08	49.63 ± 0.07	49.93 ± 0.25^{b}
T_2	49.54 ± 0.19	49.29 ± 0.15	49.06 ± 0.04	48.64 ± 0.12	49.13 ± 0.38^{c}

T_3	47.28 ± 0.04	47.04 ± 0.06	46.84 ± 0.06	46.34 ± 0.11	46.87 ± 0.4^{d}
T_4	46.77 ± 0.21	46.41 ± 0.1	45.93 ± 0.06	45.79 ± 0.02	46.22 ± 0.45^{e}
Mean	49.19 ± 2.19^{a}	48.96 ± 2.28^{b}	48.73 ± 2.4^{c}	48.38 ± 2.35^{d}	

Mean values for the hardness of ice cream

Treatment	**Days**				**Mean**
	0	**7**	**14**	**21**	
T_0	99 ± 1	99.16 ± 1.25	99 ± 1	100.16 ± 1.04	99.33 ± 0.56^{e}
T_1	105 ± 1	105.66 ± 0.57	105 ± 1	106.33 ± 0.57	105.5 ± 0.63^{d}
T_2	109 ± 1	109.33 ± 0.57	109 ± 1	110 ± 1	109.33 ± 0.47^{c}
T_3	113 ± 1	113.5 ± 0.5	113 ± 1	113.83 ± 0.28	113.33 ± 0.4^{b}
T_4	117 ± 1	117.33 ± 1.15	117 ± 1	118 ± 0	117.33 ± 0.47^{a}
Mean	108.6 ± 6.98^{b}	109 ± 7.02^{ab}	108.6 ± 6.98^{b}	109.66 ± 6.86^{a}	

Mean values for calcium content

Treatment	**Days**				**Mean**
	0	**7**	**14**	**21**	
T_0	0.44 ± 0.11	0.44 ± 9.18	0.44 ± 0.13	0.44 ± 0.11	0.44 ± 0^{e}
T_1	0.49 ± 0.11	0.49 ± 8.86	0.49 ± 0.11	0.49 ± 0.11	0.49 ± 0^{d}
T_2	0.6 ± 0.11	0.6 ± 8.37	0.6 ± 0.08	0.6 ± 0.11	0.6 ± 0^{c}
T_3	0.7 ± 0.11	0.7 ± 7.39	0.7 ± 0	0.7 ± 0	0.7 ± 0^{b}
T_4	0.74 ± 0.11	0.74 ± 5.54	0.74 ± 0.01	0.74 ± 0	0.74 ± 0^{a}

Mean	0.6 ± 0.12[a]	0.6 ± 0.12[a]	0.6 ± 0.12[a]	0.6 ± 0.12[a]	

Mean values for appearance of ice cream

Treatment	**Days**				**Mean**
	0	**7**	**14**	**21**	
T_0	8.1 ± 0.04	8.56 ± 0.05	8.53 ± 0.05	8.5 ± 0.09	8.55 ± 0.04[a]
T_1	8.6 ± 0.09	8.56 ± 0.05	8.53 ± 0.05	8.5 ± 0.09	8.55 ± 0.04 [a]
T_2	8.5 ± 0.09	8.46 ± 0.05	8.43 ± 0.05	8.43 ± 0.05	8.45 ± 0.03 [b]
T_3	8.5 ± 0.09	8.46 ± 0.05	8.43 ± 0.05	8.43 ± 0.05	8.45 ± 0.03 [b]
T_4	8.5 ± 0.09	8.46 ± 0.05	8.43 ± 0.05	8.4 ± 0.09	8.45 ± 0.04 [b]
Mean	8.54 ± 0.05 [a]	8.5 ± 0.05 [ab]	8.47 ± 0.05 [ab]	8.45 ± 0.04 [b]	

Mean values analysis for flavor of ice cream

Treatment	**Days**				**Mean**
	0	**7**	**14**	**21**	
T_0	8.53 ± 0.05	8.53 ± 0.05	8.5 ± 0.05	8.47 ± 0.02	8.51 ± 0.02[a]
T_1	8.45 ± 0.04	8.46 ± 0.02	8.41 ± 0.02	8.43 ± 0.02	8.44 ± 0.02[a]
T_2	8.3 ± 0.04	8.28 ± 0.02	8.25 ± 0.05	8.23 ± 0.03	8.26 ± 0.02[b]
T_3	8.25 ± 0.05	8.21 ± 0.02	8.18 ± 0.02	8.16 ± 0.02	8.2 ± 0.03[b]
T_4	6.5 ± 0.25	6.58 ± 0.14	5.85 ± 0.05	5.84 ± 0.05	6.19 ± 0.4[c]

Mean	8 ± 0.84[a]	8.01 ± 0.81[a]	7.84 ± 1.11[b]	7.83 ± 1.11[b]	

Mean values analysis for taste of ice cream

Treatment	**Days**				**Mean**
	0	**7**	**14**	**21**	
T_0	9 ± 0.05	8.99 ± 0.04	8.98 ± 0.04	8.97 ± 0.03	8.98 ± 0.01[a]
T_1	9 ± 0.05	8.97 ± 0.02	9.01 ± 0.02	9.03 ± 0.05	9 ± 0.02[a]
T_2	8.5 ± 0.09	8.51 ± 0.07	8.53 ± 0.05	8.46 ± 0.05	8.5 ± 0.02[b]
T_3	7.3 ± 0.39	6.9 ± 0.05	6.88 ± 0.02	6.85 ± 0.03	6.98 ± 0.21[c]
T_4	5.91 ± 0.07	5.9 ± 0.05	5.88 ± 0.03	5.86 ± 0.03	5.89 ± 0.02[d]
Mean	7.94 ± 1.32[a]	7.85 ± 1.38[ab]	7.85 ± 1.4[ab]	7.83 ± 1.41[b]	

Mean values analysis for overall acceptability of ice cream

Treatment	**Days**				**Mean**
	0	**7**	**14**	**21**	
T_0	8.5 ± 0.09	8.48 ± 0.07	8.45 ± 0.04	8.36 ± 0.02	8.45 ± 0.05[a]
T_1	8.6 ± 0.09	8.6 ± 0.04	8.55 ± 0.04	8.45 ± 0.1	8.55 ± 0.07[a]
T_2	8.45 ± 0.1	8.5 ± 0.05	8.45 ± 0.04	8.36 ± 0.07	8.44 ± 0.05[a]
T_3	6.6 ± 0.1	6.58 ± 0.07	6.55 ± 0.04	6.19 ± 0.57	6.48 ± 0.19[b]
T_4	5.5 ± 0.09	5.48 ± 0.07	5.45 ± 0.04	5.42 ± 0.02	5.46 ± 0.03[c]
Mean	7.53 ± 1.4[a]	7.53 ± 1.42[a]	7.49 ± 1.41[ab]	7.36 ± 1.44[b]	

Discussion

Skim milk powder is ideal ingredient for the formation of ice cream as it contributes to the total number of solids in milk. In this study, powdered form of skim milk was purchased from shopping mart located in Faisalabad. The proximate composition is elaborated in the table (a). Results of the proximate composition shows that the powder form of milk contains about 31% protein, about 96% milk solids not fat, fat composition of 0.5%, ash 8.5%, moisture 3.5%, and the total solids are up to 97%. These findings also correlate with the results of (Murtaza *et al.*, 2015).Cream is also an essential ingredient of ice cream and gives ice cream a smooth structure. The proximate analysis including protein, fat, ash, moisture and other factors MSNF and total solid were determined.The proximate analysis of eggshell was also done and the table (a) shows the protein value 4%, moisture 2.5%, fat 1% and mineral value 92.5%. The calculated calcium value also resembles with the study of Khan *et al.* (2020).

The amount of protein in eggshell ice cream reduced from 4.29% to 3.26%. Sample of ice cream without the addition of eggshell had highest protein content than compared to the 3.1% found in the T_4. Eggshell was replaced by skim milk powder with 40% concentration in T_4 treatment. The maximum protein content was observed in T_0 which is 4.29%. The samples T_1, T_2, T_3, and T_4 had protein values as 4.07%, 3.73%, 3.53%, and 3.26% respectively. It is concluded from this observation that protein value decreased in the treatments as the concentration of eggshell powder increased in the ice cream samples.The range of fat content in the ice cream was reduced from 11.73% to 10.15%. The maximum fat content was observed in the controlled ice cream sample named as T_0 which is 11.73% and the noted minimum fat value was recorded in the sample named as T_4 which is 10.15%. The reason of minimum fat value in T_4 may be that it had highest amount of eggshell powder as a replacement to skim milk.The maximum value of ash was found in T_4 which is 1.2% and minimum was observed in T_0 which is 0.77%. The other samples of eggshell added ice cream T_1, T_2, and T_3 had 0.88%, 1.02%, and 1.12 ash content respectively.

The maximum value noted for the moisture was in the treatment T_0 having 64.5% water content. and the minimum value observed was in the sample named as T_4 having 63.5% water content. This slight change in water content was due to the increased concentration of eggshell powder. The samples T_1 has 64.34%, T_2 has 64.2%, T_3 has 64.1%, and T_4 has 63.94% moisture content.The maximum value for TSS was observed in T_0 which is 30.5 and the minimum was in T_4 with 23.95. the reason for the decrease of TSS in sample maybe the increased concentrations of eggshell powder. As eggshell powder does not completely dissolve. The other eggshell added ice cream samples T_1, T_2, and T_3 had 28.66, 25.41, and 25.04 brix respectively.The pH values decreased from 6.68 to 6.4. The table shows that pH values followed a decreasing trend from T_0 to T_4. Eggshell itself does not have a pH value but its trend is observed towards acidic behavior when fortified. The maximum pH value was observed in T_0 which is 6.68 and minimum was observed in T_4 which is 6.4. Other eggshell added ice cream samples T_1, T_2, and T_3 had 6.48, 6.49, and 6.42 pH values respectively. The maximum acidity was noted in T_4which is 0.22 and the minimum value was observed in T_0 which is 0.18. T_1 and T_2 almost had the same acidity values of 0.2. The sample T_3 of eggshell added ice cream had 0.21 titratable acidity value.

The minimum melting time duration was noted in T_0 which is 25.1 minutes and the maximum duration was noted in T_4 which is 27.97. This means that the treatment with the maximum concentration of eggshell powder showed more resistance towards melting. Other eggshell added ice cream samples T_1, T_2, and T_3 had mean values 25.62, 27.04, and 27.47 minutes

respectively. The range of viscosity from T_0 to T_4 is illustrated in the table. The treatment T_0 exhibits the minimum viscosity 65.54 cp while the treatment T_4 exhibits the maximum range of viscosity which is 73.99. Other eggshell added ice cream samples T_1, T_2, and T_3 had 71.27, 67.98, and 73.48 viscosity values respectively.It shows that the overrun gradually decreased from T_0 to T_4. The maximum overrun was observed in T_0 which is 51.9%. The minimum overrun was observed inT_4 which is 46.22%. Other eggshell added ice cream samples T_1, T_2, and T_3 had overrun values 49.93%, 49.13%, and 46.87% respectively. The decrease in the overrun might be due to the decrease in fat content when different concentrations of eggshell powder were added.

The values of the hardness varies from T_0 to T_4. The values are calculated in mm/5sec. The maximum hardness was calculated in T_4 which is 117.33 and the minimum value observed was 99.33 in treatment T_0. Other eggshell added ice cream samples T_1, T_2, and T_3 had hardness values 105.5, 109.33, 113.33 respectively. It means that the hardness of the ice cream decreased with each treatment and it can be concluded that eggshell resulted in the decrease of firm structure of ice cream.The values increased in each sample as the concentration of eggshell powder was increased in every other sample. The maximum value of calcium was calculated in T_4 which is 740mg/100g means 0.74% and the minimum calcium was calculated in T_0 which is 440mg/100g means 0.44%. In sample T_1 skim milk was replaced with 10% eggshell powder and the final calcium content obtained was 490mg/100g means 0.49%. Remaining samples T_2 and T_3 had calcium content 600mg/100g (0.6%) and 700mg/ml (0.7%) respectively. The reason for increased value of calcium in T_4 is higher concentration eggshell powder used. These results also correlate with the study of El-Shibiny *et al.* (2018) where he added Nano-sized eggshell powder in yogurt and observed the same highly significant effect as the calcium values were increased in each sample.

It can be seen clearly that there was significant effect on the appearance of ice cream with increased concentration of eggshell powder. The samples T_1 and T_2 had quite similar results and obtained 8.55 overall score. It means that these two samples were more acceptable in terms of appearance. Remaining samples T_2, T_3, and T_4 also got the same score and the obtained overall score was 8.45. It means that they were also acceptable in terms of appearance but got scores less that T_1 and T_2. So, it means that they were less acceptable.Treatment T_0 and T_1 had more acceptability than other samples for the eggshell powder ice cream. Ice cream samples showed a significant decrease in scores as the concentration of eggshell powder increased. During the storage, there was a slight decrease in the scores as there was a non-significant change in flavor during the storage. The scores ranged from 8.51 to 6.19. Samples T_0 and T_1 had a non-significant relation and the scores obtained were 8.51 and 8.44 respectively. The other two samples T_2 and T_3 got the scores 8.26 and 8.2 respectively and these two samples also had a non-significant relationship.

After increasing the concentration of eggshell powder in each treatment, the taste of the ice cream decreased. Treatment T_4 got the least score as the grainy texture of eggshell powder was being felt. On the other hand, the control sample T_1 had the highest taste score. And between the eggshell added treatments the most desirable samples were T_0 and T_1. The range of the scores obtained were from 9 to 5.89. The samples T_0 and T_1 had a non-significant relationship and obtained 8.98 and 9 scores respectively. At 0 days' storage, the overall acceptability of ice cream decreased with the increased concentration of eggshell powder. The order of acceptability is T_1, T_0, T_2, T_3, and T_4. After the storage of 21 days, the effect on overall acceptability was non-significant. The maximum score was obtained by T_1 which is 8.55 and the minimum score was obtained by T_4 which is 5.46. The second sample in order of acceptability was T_0 with score 8.45.

Third sample in terms of acceptability was T_2 with score 8.44.

Conclusions and Recommendations

According to the findings, the addition of eggshell powder to ice cream preparation improved the sensory and nutritional value of the final product. The addition of different concentrations of eggshell powder to ice cream samples resulted in an increase in calcium content and overall acceptability of the samples. There are a few recommendations listed below.

- Eggshell should be used as a source of calcium in food products.
- The manufacturing of ice cream at commercial level should begin to curtail calcium deficiency as the graininess of eggshell can be masked off with sprinkles or addition of crunchy food additives.
- There is a huge potential of utilization of eggshell powder in other products too with better quality and acceptability.

References

Aditya, S., J. Stephen and M. Radhakrishnan. 2021. Utilization of eggshell waste in calcium-fortified foods and other industrial applications: A review. Trends Food Sci. Technol. 115:422-432.

AOAC. 2016. The official methods of analysis of association of official analytical chemist international (20thed). The association of official analytical chemist. Arlington, United States of America

Civille, G.V. and K.N. Oftedal. 2012. Sensory evaluation techniques. Physiol. Behav. 107:598605.

Costa, F., J. Resende, L. Abreu and H. Goff. 2008. Effect of calcium chloride addition on ice cream structure and quality. J. Dairy Sci. 91:2165-2174

El-Shibiny, S., M.a.E.-K.M.A. El-Gawad, F.M. Assem and S.M. El-Sayed. 2018. The use of nano-sized eggshell powder for calcium fortification of cows and buffalos milk yogurts. Acta Sci. Pol. Technol. Aliment. 17:37-49

Goff, H.D. and R.W. Hartel. 2013. Ice cream structure. Ice cream. Arlington, United States of America. 313-352

Hanafi, F., N. Kamaruding and S. Shaharuddin. 2022. Influence of coconut residue dietary fiber on physicochemical, probiotic (*Lactobacillus plantarum*) survivability and sensory attributes of probiotic ice cream. LWT. 154:112725

Ismail, H.A., A.M. Hameed, M.M. Refaey, A. Sayqal and A.A. Aly. 2020. Rheological, physio-chemical and organoleptic characteristics of ice cream enriched with Doum syrup and pomegranate peel. Arab. J. Chem. 13:7346-7356

Khan, F.A., K. Ameer, M.A. Qaiser, I. Pasha, Q. Mahmood, F.M. Anjum, A. Riaz and R.M. Amir. 2020. Development and analysis of bread fortified with calcium extracted from chicken eggshells of Pakistani market. Food Sci. Technol. 41:14-20.

Murtaza, M.A., N. Huma, I. Mustafa, H. Nawaz, M.S. Murtaza and M.I. Ullah. 2015. Composition and microstructure of buffalo skim milk powder as influenced by concentration and drying temperatures. Pak. J. Zool. 47:1157-1161.

Sofjan, R.P. and R.W. Hartel. 2004. Effects of overrun on structural and physical characteristics

of ice cream. Int. J. Dairy Technol. 14:255-262.