

Formulation and Characterization of Eggshell Calcite Powder

S. Kalaipriya*¹ and R. Radha²

Received: 08 Feb 2021 | Revised accepted: 03 Apr 2021 | Published online: 07 Apr 2021

© CARAS (Centre for Advanced Research in Agricultural Sciences) 2021

ABSTRACT

Calcium is an essential nutrient to maintain bone health. Eggshell which is wasted in high amounts, is an excellent source of calcium carbonate which can be used as supplementation purposes. The eggshells are collected from the same batch of hens which are fed uniformly. Then eggshells are washed, powdered with mixer grinder, dried in hot air oven and processed in muffle furnace at 900°C for 4 hours. Further FE SEM analysis, Calcium analysis, Heavy metal analysis, Ash content, moisture content analysis was done. The FE SEM analysis showed results showed that the calcite powder was roughly spherical in shape, 157-81 nano meter in size. calcite powder contains 97% of calcium with no traces of heavy metals and zero moisture content. Since the developed calcite powder contains more calcium it can be used as a calcium supplement.

Key words: Eggshell, Calcite, Calcium, Bone health

Calcium is an essential mineral required to maintain healthy bones, teeth, heart, muscles and nerves in the human body. The calcium requirement infants are 500 mg, Children of year 1 to 9 is 600 mg, Adolescence is 800 mg, Calcium requirement of adult men and women is 600 mg, except for pregnant and lactating women where their requirement is 1200 mg [1]. Osteoporosis is a condition where the bone loses its density and becomes weak and brittle. About 50 million people in India are either osteoporotic or have low bone mass [2]. Inadequate nutrition and lack of exposure to sunlight is the important cause of Osteoporosis.

When the calcium requirements are not met through the diet calcium supplements are recommended. Calcium supplements are in different forms like calcium carbonate, calcium citrate, calcium lactate and calcium gluconate which are composed of 40%, 21%, 13%, 9% of elemental calcium respectively. Calcium supplementation can also be done with eggshell, as eggshells are natural source of calcium which is composed of 40 percent calcium carbonate [3]. Compared to pure calcium carbonate, calcium absorption from eggshells was 64% greater [4].

MATERIALS AND METHODS

Collection of eggshells

A massive amount of eggs shells and membrane are wasted annually, eggs are utilized for various purposes and the shells are thrown away. These shells can be utilized for various purposes like adsorbents, antimicrobial agents, catalysts, calcium supplementation as they contain calcium

carbonate [5]. The chicken eggshells are collected from the egg product industry. The chicken eggs shells were collected from the same batch where hens were uniformly fed. The collected eggshells are washed properly and then dried. The dried eggshell powder is stored in air tight container.

Processing of eggshell powder

The eggshell powder is washed thoroughly for the removal of eggshell membrane and other impurities if any. The eggshell powder is washed with the purified water with less than 10 ppm and left for half an hour in the water for the eggshell powder to settle down, other impurities and membrane would be separated and it would form a froth and the eggshell powder is filtered. This process is repeated several times till the water remains pure of its nature which represents the impurities are being washed out.

The keeping quality of food is greatly affected by the moisture content present in it as moisture helps in growth of microorganisms resulting in spoilage of the product [6]. So, the wet eggshell powder is completely dried using hot air oven at 135°C for about 3-4 hours. And the dried powder was stored in sterilized container.

Development of eggshell calcite powder

Almost 94-96% of an eggshell is reported as CaCO_3 [7-8]. The dried powder is weighed and noted. Then the eggshell powder is taken in 6 crucibles as 6 samples and heated in the muffle furnace. At the beginning the temperature were kept at 200°C for half an hour then one sample is taken to examine the physical change. Then the temperature is raised to 400°C and maintained at half an hour and the 2nd sample is taken to examine the physical change. Then the temperature was raised to 600°C for half an hour then sample 3 is taken to examine the physical change. At 800°C for half an hour, the sample 4 was taken out. Sample 5 was taken to examine the physical change at 900°C for an hour. Finally, the sample 6

*S. Kalaipriya

priya80000@gmail.com

¹⁻²Avinashilingam Institute for Home Science and Higher Education for Women, Coimbatore -641 043, Tamil Nadu

were kept at 900°C for 4 hours and then the samples are collected and weighed.

Characterization of developed eggshell powder

FE SEM: Unlike other microscopes Field Emission Scanning Electron Microscope works with electrons not with light. It works on the principle of electrons liberated from the emission source of the device which helps in visualisation of the object [9]. Initially gold sputtering is done on developed eggshell powder and then it is Scanned in Field emission scanning electron microscope. The electrons liberated from the emission source. The electrons bombards the developed eggshell powder and bombards. Due to these secondary electrons are emitted. The detector produces electric signals by capturing those secondary electrons. This electrical signal results in production scan images in the monitor which can be saved.

Estimation of calcium: The sample was dissolved in hydrochloric acid and made upto the 100 ml. 10 ml of the ash solution was pipetted out in a conical flask and 90 ml of distilled water was added to it. Added 2 drops of methyl red indicator. It was made strongly alkaline by adding ammonia and kept for boiling. 20 ml of saturated ammonium oxalate was added to the solution, 10 ml each time to ensure complete precipitation directly. When it was hot, a few drops of acetic acid was added to render the medium acidic. The precipitate was allowed to settle overnight. The next morning the solution was filtered with Whatman No.40 filter paper. The precipitate was washed first with ammoniacal water and then with hot water several times until it was free from chloride. To test it 5 ml of the washing was collected, in a test tube and a drop of silver nitrate solution was added. The washing was continued till there was no precipitate with silver nitrate or calcium chloride solution. The filter paper was collected in a flask by making a hole in the filter paper. To this, 2 ml of 2N sulphuric acid was added. This solution was heated to 60-80°C and when still hot was titrated against N/100 potassium

permanganate solution. From the volume of potassium permanganate solution used up the milligrams of calcium present in 100g of the sample was calculated.

Heavy metal analysis: To find the toxicity of the eggshell powder the heavy metal analysis were done. Mercury, Lead, cadmium and arsenic were tested with the AOAC 15.01 method.

Determination of moisture content: Heated a pair of weighing bottles at 100°C in an oven and labelled A and B. Placed on an asbestos sheet for 2 minutes and then transferred them to a desiccator where they remained for half an hour. Developed eggshell powder was placed and their weights in an analytical balance. Repeated this procedure till two successive weights obtained were constant (with maximum difference of 0.0002g). Weighed definite amounts of developed eggshell powder in each dish and placed in an electric oven and heated for 2 hours at 150°C and weighed. And the difference gave the moisture content of developed eggshell powder.

Determination of ash content: About 5g of the developed eggshell Powder was weighed and placed in crucible (which had previously been heated to about 600°C and cooled). The crucible was then heated till all the material was completely charred, followed by heating in a muffle furnace for about 3-5 hours, at 600°C. The crucible was then cooled in a desiccator and weighed.

RESULTS AND DISCUSSION

Colour and texture

The eggshell powder was taken out at different temperature for observation, at different temperature the colour varied and at the final stage the eggshell calcite was white in colour. The eggshell calcite powder was like fine crystalline powder.



Fig 1 At 200°C



Fig 2 At 400°C



Fig 3 At 600°C



Fig 4 At 800°C



Fig 5 At 900°C

Size and surface morphology

To know the size and surface morphology the eggshell was viewed under field emission scanning electron microscope. The eggshell powder and the developed eggshell calcite was compared and analyzed. The (Fig 1-5) are eggshell powders and eggshell calcite powder respectively. The (Fig 1-5) the eggshell powder is viewed with the working distance of

9.85 mm and at 5.0kV, the size every particle falls between 40–100 micro meter. And the (Fig 6-7) was viewed at working distance of 5.93 mm at 5 Kv it showed that the particles of eggshell calcite powder are lies between the 157-81 nano mater in size. The eggshell powder is roughly crystalline in structure as shown in the (Fig 6). The eggshell calcite powder is roughly spherical in shape as shown in the (Fig 7).

Table 1 Estimation of heavy metals

Test parameters	Test result - eggshell powder	Test result - eggshell calcite powder
Lead	BDL (DL:0.1 mg/kg)	BDL (DL:0.1 mg/kg)
Cadmium	BDL (DL:0.03 mg/kg)	BDL (DL:0.03 mg/kg)
Mercury	BDL (DL:0.1 mg/kg)	BDL (DL:0.1 mg/kg)
Arsenic	BDL (DL:0.05 mg/kg)	BDL (DL:0.05 mg/kg)

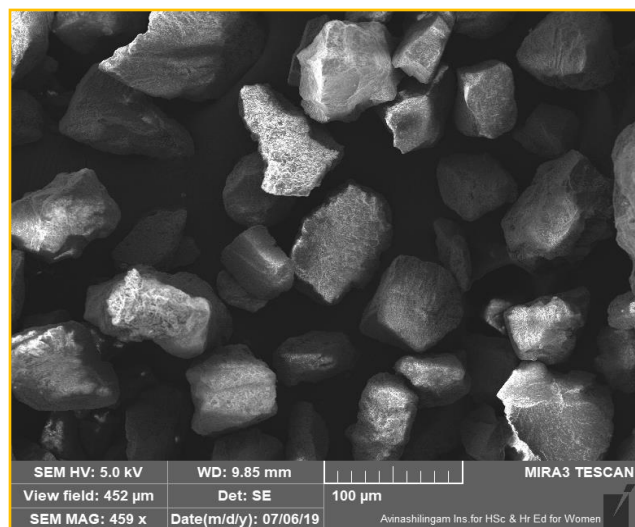


Fig 6 Shape of calcite

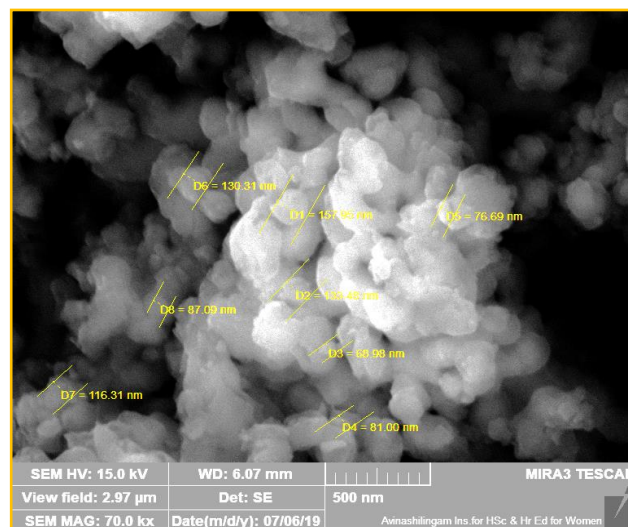


Fig 7 Size of calcite

Calcium analysis of eggshell calcite powder

Calcium estimation was done based on the AOAC method. And it was estimated that there is 97% of Calcium in the eggshell calcite powder.

Heavy metal analysis

Heavy metal analysis was done based on the AOAC.15.01. Lead, Cadmium, Mercury, Arsenic was analyzed and the result showed that all the heavy metals were below the detectable limit of both the eggshell powder and eggshell calcite powder.

Determination of moisture

Moisture content was determined by AOAC method and found the eggshell calcite powder contains 0% moisture.

Determination of ash

The ash content of Eggshell powder was determined with AOAC method and the eggshell contains 53.93% ash.

CONCLUSION

From the above study it is concluded that the eggshell powder is heated in the muffle furnace at constant temperatures and converted into eggshell calcite powder (a polymorph form of calcium) which is identified as nano spherical particle, it is free from heavy metals and it contains 97% of calcium, which can be used as a natural source of calcium supplementation.

LITERATURE CITED

1. ICMR. 2010. Indian Council of Medical Research, 2010.
2. Mithal A, Kaur P. 2012. Osteoporosis in Asia: A call to action. *Cure Osteoporosis Rep.* 10: 245-247.
3. Brun LR, Lupo M, Delorenzi DA, Di Loreto VE, Rigalli A. 2013. Chicken eggshell as suitable calcium source at home. *International Journal of Food Sciences and Nutrition* 64(6): 740-743.
4. Daengprok W, Garnjanagoonchorn W, Naivikul O, Pornsinlpatip P, Issigonis K, Mine Y. 2003. Chicken eggshell matrix proteins enhance calcium transport in the human intestinal epithelial cells, Caco-2. *Journal of Agricultural and Food Chemistry* 51(20): 6056-6061.
5. Ahmed TA, Kulshreshtha G, Hincke MT. 2019. Value-added uses of eggshell and eggshell membranes. In: *Eggs as Functional Foods and Nutraceuticals for Human Health*. pp 359-397.
6. Anonymous. 2012. Department of Food, Nutrition, and Packaging Science. Clemson University.
7. Zaman T, Mostari M, Mahmood MAA, Rahman MS. 2018. Evolution and characterization of eggshell as a potential candidate of raw material. *Cerâmica* 64(370): 236-241.
8. Neunzehn J, Szuwart T, Wiesmann HP. 2015. Eggshells as natural calcium carbonate source in combination with hyaluronan as beneficial additives for bone graft materials, an in vitro study. *Head and Face Medicine* 11(1): 1-10.
9. Chalmers GR, Bustin RM, Power IM. 2012. Characterization of gas shale pore systems by porosimetry, pycnometry, surface area, and field emission scanning electron microscopy/transmission electron microscopy image analyses: Examples from the Barnett, Woodford, Haynesville, Marcellus, and Doig units Characterization of Gas Shale Pore Systems. *AAPG Bulletin* 96(6): 1099-1119.