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# Recycling of Chicken Egg Shells into Nanopowder: Synthesis, and its Properties

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## Abstract:
Increase in unconventional resources of calcium ($Ca^{+2}$) for fowls, aquaculture and native animals was improved. This work was planned to define the most polymorph of calcium carbonate ($CaCO_3$) that take place in the two types of chicken eggshells (local and imported type). In this research, the comparative analysis of calcium carbonate ($CaCO_3$) content was approved for nominated eggshells of native strain and imported chicken via Field Emission Scanning Electron Microscope (FESEM), Transmission Electron Microscope (TEM), Fourier-Transform Infrared Spectroscopy (FTIR) and Powder X-Ray Diffraction (PXRD) analysis. The results demonstrate that native and imported chicken eggshells comprise calcite morph that had shape between rhombohedral and spherical with distinguished pores distribution in the surface and crystallization size (31) nanometer for local chicken eggshells and (32) nanometer for import chicken eggshells respectively. The authors brief their results that local and import chicken eggshells had the top resources of calcium carbonate ($CaCO_3$).

**Key words**: Chicken egg shells, EM, Nanopowder, Production, Physiochemical analysis, Reusing.

## Introduction:
Chicken eggshells are discarded resources from native bases for example hatcheries, homes and fast food productions [1] and are liable to be freely collected adequately. Eggshells discarded clearance ads to conservational contamination. Challenges related with discarding of eggshells consist of cost, accessibility of throwing away places, odour, flies and harshness [2]. Furthermore, chicken eggshells discarded treating is presently produced at huge quantities of numerous tons per day [3]. Furthermost eggshell remaining is thrown away since additional treating is too costly and burdensome [4]. Numerous researchers have been observed for methods to use the eggshells discarded, such as, via eggshells powder as a steadying material for cultivating soil properties, Ultraviolet-protecting essences [5, 6] and as a basis of Calcium in human diet [7].

$Ca^{+2}$ measures comprise carbonate, citrate, or gluconate salts that are not every time in effect [8]. For that reason, ordinary bases of minerals and vitamins are becoming further prevalent [9]. Eggshell $Ca^{+2}$ is the superlative ordinary basis of calcium and it is around 90% absorbable than oyster shells, limestone or coral bases [10, 11]. The structure of an eggshell is exactly like that of our bones and teeth [10, 12].

$CaCO_3$, a pharmaceutical excipient, is principally recycled as diluent in solid dose formulae. It is furthermore recycled as a base for medicinal and dental preparations, buffering and dissolution assistance in dispersible tablets, in addition to food chemical addition and calcium complement [13, 14, 15, 16].

Currently, there is an excessive attention in the discovery of novel unpolluted $CaCO_3$ sources. $CaCO_3$, gained from bones flour, does not comprise similar bioavailability of $Ca^{+2}$ gotten from artificial bases. $CaCO_3$ from eggshells has a benefit for not having toxically rudiments [17].

Conferring to this information, a novel skill of eggshells treating was established. Egg productions harvest excessive quantity of shells and their concluding purpose is a conservational effect challenge. This research is planned to organize and estimate $CaCO_3$ from egg shell as it has a probable presentation in the development of a novel opportunity of pharmaceutical excipient, bone scaffold development, teeth manufacturing and drug carrier and delivery. The absence of data on these topics advises that there is a hopeful area to be discovered [18], and these resources will permit quick progresses in relative studies of the organic elements of chicken eggshells and their practical consequences [13].

The main objective of this study is to discover an ecologically and economically interesting usage of discarded chicken eggshells by rotating them into nanoparticles that can be recycled as steadying material for refining soil properties, to progress of a novel choice of pharmaceutical excipient, bone scaffold development, teeth manufacturing and drug carrier and delivery. The particular objective of this study is to explore the physical-chemical of CaCO3 gotten from diverse bases of chicken egg shells.

## Materials and Methods:

The chicken eggs (Gallus gallus) of the different sources (local and import (Turkey origin)) (selected the white color one) were bought from Mosul markets, then weighted on a weighing balance (Sartorius AG Gottikàen, GP5202, d=0.01g, Germany), broken into a beaker. The raw eggshells were washed under tap water till the egg albumen was totally removed and peel off all of the shell's membranes from inside the shells. The shells were washed with deionized water then wrapped with a toilet paper to dry the water contented. Lastly the shells were weighed and ground into a fine powder using blender, weighted, then sieved through $\leq 75$ μm sieve. The sieved powder was additionally dehydrated in the lab oven at fifty Celsius degree for 120 hours and converted into nanoparticles using a mechanical method in the existence of Ball mill (Wisd® Ball Mill, Korea) for 7days then kept at fifty Celsius degree in a sterilized bottle previous to usage [19, 20].

### Chicken eggshells description

### Electron Microscopy (EM)

Analysis of prepared nanoparticles was done by FESEM tracked by image exploration with their particular software correspondingly and a transmission electron microscope (TEM) (Hitachi H-7100, Japan) for detecting the shape and size of the nanoparticles. For FESEM analysis, the samples were individually prepared, covered with gold and viewed [21, 22]. The chicken eggshells nano powder was liquefied in ninety nine percentage ethanol and sonicated for thirty mins. (1-3) droplets of suspension was loaded on top of the carbon-enclosed copper grid and positioned on a filter paper to dehydrate at lab temperature previously observing by TEM [23, 24].

### FTIR analysis

The biochemical tests were completed by FTIR in a variety of two hundred and eighty to four thousands $cm^{-1}$ at a determination of two $cm^{-1}$ and by a scan speediness of sixty four/s. The tablets of the nanoparticles chicken eggshells powders were examined separately in a mass percentage of one weight percentage in Kbr powder, and tests were achieved over Ultra Attenuated Total Reflection (UATR) methods [25, 26].

### PXRD examination

One to two grams of powder of two form of eggshells were recycled for this examination. The examination required the performance of extensive-direction XRD to distinguish the nature and crystal-like apparatuses of the examples. All crystal-like stages existing were documented using XRD analyzer (Angstrom Advanced Inc. ADX-2700, X-Ray Powder diffraction Instrument) over the use of CuKα (λ=1.540562 Å) at forty kV and thirty mA [27, 28]. The deflection formula was calm at a speed of perusing 0.02 degrees/s in 2θ at a variety of (5° - 60°) at $37^oC$. XRD by Scherrer's formulation to match with the standards gotten from Transmission Electron Microscopy examination. The crystal-like sizes (Dv) is an informal procedure that flexibly differentiates among straining and dimension made peak lengthening overseeing the peak measurement as a meaning of Two theta [29].

## Results:

The study involved recycling of chicken eggshells (local and import) and proportional examination of $CaCO_3$ contented in them, which contain percentage method of analysis via using (7) chicken eggs of each type (Table 1and 2).

**Table 1. Percentage analysis of micropowder of (7) local chicken eggshells.**

| No. of sample | whole weight of local chicken egg (g) | whole weight of local chicken eggshell (g) | Percentage of whole weight of local chicken eggshell (g)/ whole weight of local chicken egg (g) | whole weight of local chicken eggshell micropowder (g) / whole weight (g) | Percentage of whole weight of local chicken eggshell micropowder (g) / whole weight of local chicken egg (g) |
|---|---|---|---|---|---|
| 1 | 48.01 | 5.64 | 11.75 | 5.55 | 98.404 |
| 2 | 48.78 | 5.43 | 11.132 | 5.35 | 98.527 |
| 3 | 45.73 | 5.51 | 12.049 | 5.46 | 99.093 |
| 4 | 50.58 | 5.91 | 11.684 | 5.79 | 97.97 |
| 5 | 51.45 | 5.7 | 11.079 | 5.52 | 96.842 |
| 6 | 51.69 | 5.54 | 10.718 | 5.49 | 99.097 |
| 7 | 48.34 | 5.36 | 11.088 | 5.11 | 95.335 |
| Average | 49.23 | 5.58 | 11.36 | 5.47 | 97.90 |

**Table 2. Percentage analysis of micropowder of import (7) chicken eggshells.**

| No. of sample | whole weight of import chicken egg (g) | whole weight of import chicken eggshell (g) | Percentage of whole weight of import chicken eggshell (g)/ whole weight of import chicken egg (g) | whole weight of import chicken eggshell micropowder (g) / whole weight (g) | Percentage of whole weight of import chicken eggshell micropowder (g) / whole weight of import chicken egg (g) |
|---|---|---|---|---|---|
| 1 | 68.22 | 7.28 | 10.671 | 6.79 | 93.27 |
| 2 | 70.74 | 6.7 | 9.471 | 6.6 | 98.51 |
| 3 | 66.78 | 6.62 | 9.913 | 6.52 | 98.9 |
| 4 | 66.42 | 6.32 | 9.515 | 6.26 | 99.1 |
| 5 | 69.07 | 6.65 | 9.628 | 6.52 | 98.05 |
| 6 | 66.43 | 6.39 | 9.619 | 6.29 | 98.44 |
| 7 | 66.12 | 5.93 | 8.969 | 5.84 | 98.482 |
| Average | 67.68 | 6.56 | 9.68 | 6.40 | 97.82 |

Physical picture data (texture, color and hardness) of local and import chicken eggshells are existing in Table 3.

**Table 3. Physical description of local and import chicken eggshells.**

| Type of eggshell | Physical characterization | | |
|---|---|---|---|
| | Texture | Color | Hardness |
| Local chicken eggshells | Very smooth | off-white | Hard |
| Import chicken eggshells | Smooth | White | Semi-hard |

**Description of chicken eggshells nanoparticles**

The FESEM and TEM for nanoparticles shown calcite calcium carbonate nanoparticles with shape between rhombohedral and spherical with distinguished pores distribution in the surface but the spherical shape is dominates than the other shape and with a regular size of ≤100 nm for FESEM (Fig. 1) and with variety size of ≤50 nm (Fig. 2) for TEM. There was no modification in the elemental structures of the gotten $CaCO_3$ calcite nanoparticles powder after production using of roller mill method. This reveals the roll milling benefits in the breakdown of the larger sized calcite rods into smaller spherical ones with pores on their surfaces.

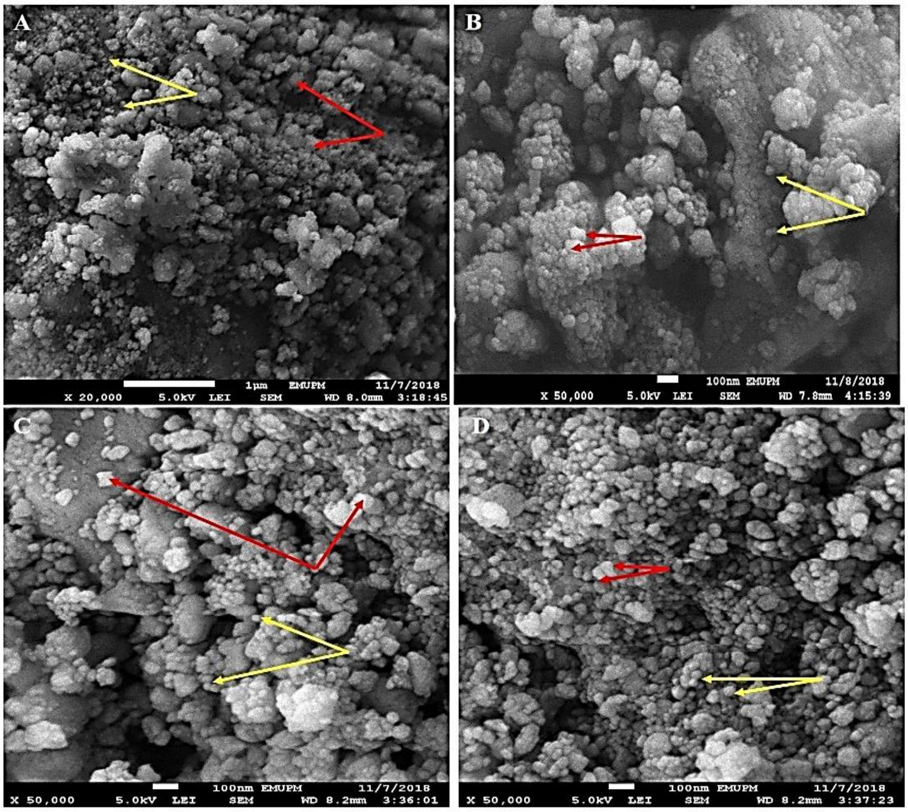


**Figure 1. FESEM micrographs of local (A&B) and import (C&D) chicken eggshells. The FESEM micrograph shows that both kinds of chicken eggshells have construction between rhombohedral (red arrows) and spherical (yellow arrows) shape of particle size, magnification of 50000 X.**

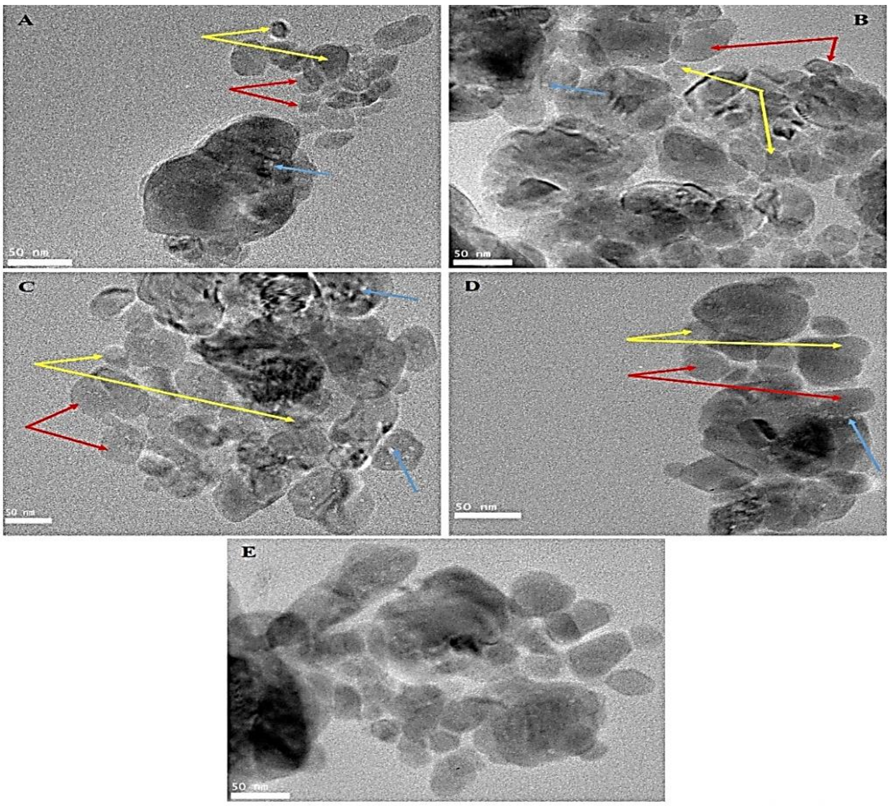


**Figure 2. TEM micrographs of local (A&B) and import (C, D &E) chicken eggshells. The TEM micrograph shows that both kinds of chicken eggshells have construction between rhombohedral (red arrows) and spherical (yellow arrows) shape crystal with hollows on their surfaces (blue arrows), magnification of 50 nm.**

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**FTIR investigation**

FTIR bands and variety of the main bands of the chicken eggshells nano powder are presented in (Fig. 3). In both kinds of chicken eggshells, different design of spectra was detected. The bands characterize some main sets demonstrating the stretching of hydroxyl, carbonyl and oxygen groups. The existence of C-O, C=O, C=C and Ca-CO groups of the calcite bands of $CaCO_3$ were also described. The absorbance spectrum of $CaCO_3$ nano powder presented bands at 1402.82-1405.59 cm−1, 873.18-873.72 cm-1 and 710.38-710.72 cm-1 for both kind of chicken eggshells due to carbonate ($CO_3$).

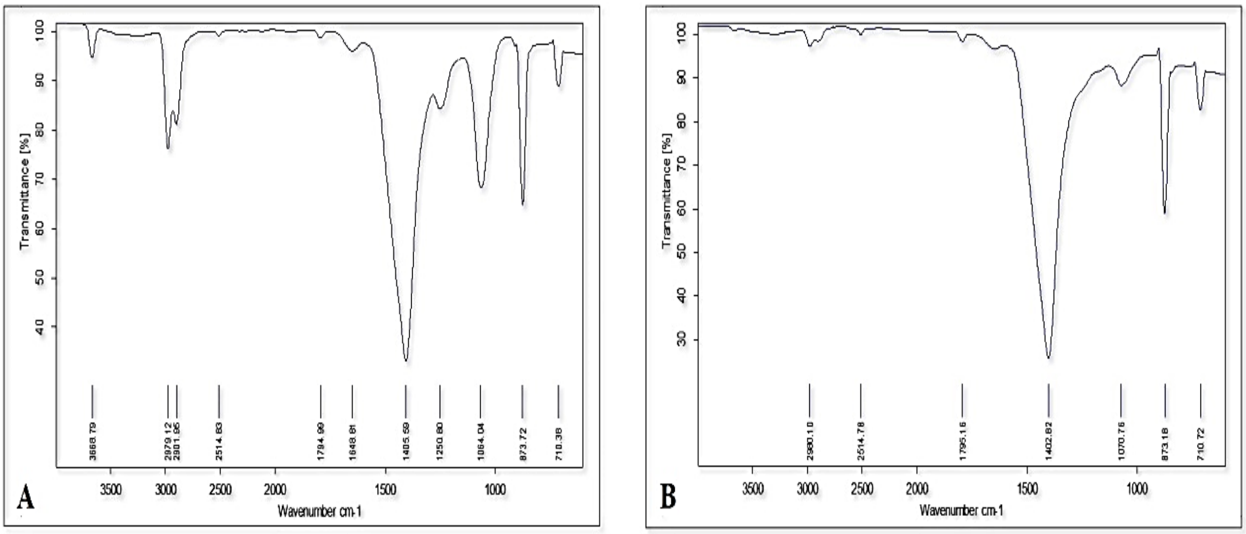


**Figure 3. FTIR analysis of local (A) and import (B) chicken eggshells.**

**PXRD investigation**

PXRD outline of the ready chicken eggshells nano powder is presented in (Fig. 4). The XRD examination is a penetrating assessment intended for the acknowledgement of crystal-like phases of mineral combinations. Crystallinity phases were determined with diffraction angles from 5-60° at thirty seven Celsius degree. Fig. 4 indicates that PXRD captivation peaks in the nano powders harmonized each other. This indicate that the crystal-like value of calcite calcium carbonate nanoparticles in these concentrates is retained during the procedure of manufacture. Four noticeable peaks established each in $CaCO_3$ calcite nanoparticles powder bands. The primary set of peaks were in two theta= 23.08°-29.357°, while the additional set were in two theta=31.436°-39.447° and the other set were in two theta=43.195°-48.55° and last set were in two theta=56.555°-58.141° (Table 4 and 5). This indicate that the crystal-like value of $CaCO_3$ calcite nanoparticles powder was preserved during the procedure of manufacture. These results are associated to a typical $CaCO_3$ structure ICDD-card number 01-083-1762. The PXRD examination done presented vital data on crystal phase of the ingredients present in the eggshells. The obtained data from the bands recognized the presence of the characteristic peaks of calcite. PXRD and TEM approaches were castoff to examine the phase outline, the shape and dimension of prepared calcium carbonate concentrate. PXRD outlines of eggshells concentrates are similar and show several deflection topics that might be assigned to rhombohedral and spherical crystal-like construction with crystallite size 31and 32 nm for local and import chicken eggshell, respectively.

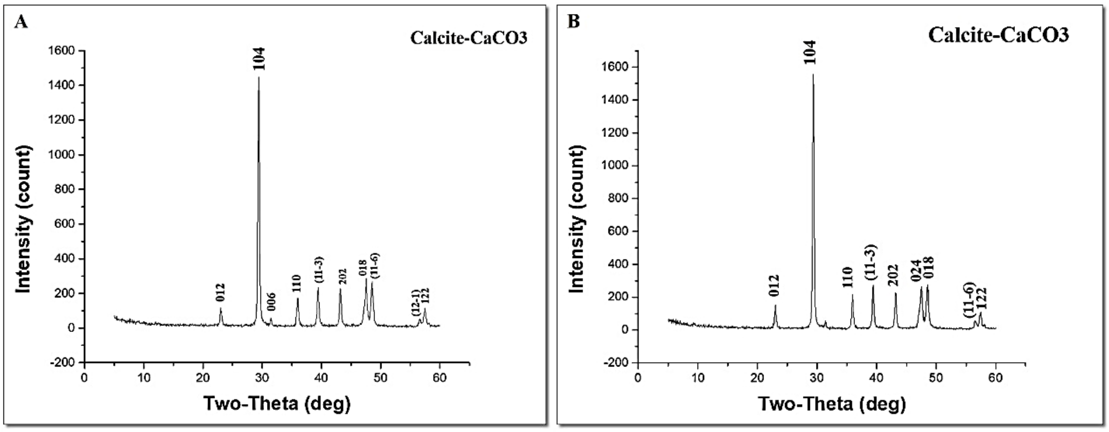


**Figure 4. PXRD analysis of local (A) and import (B) chicken eggshells.**

**Table 4. PXRD analysis of nanopowder of (7) local chicken eggshells.**

| 2-Theta | d(nm) | (h | k | l) | BG | Height | I% | Area | I% | FWHM | XS(nm) |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 23 | 0.38636 | (0 | 1 | 2) | 9 | 97 | 6.8 | 658 | 6.6 | 0.288 | 28 |
| 29.4 | 0.30355 | (1 | 0 | 4) | 12 | 1426 | 100 | 10019 | 100 | 0.299 | 27 |
| 31.449 | 0.28423 | (0 | 0 | 6) | 13 | 37 | 2.6 | 143 | 1.4 | 0.164 | 51 |
| 35.995 | 0.2493 | (1 | 1 | 0) | 10 | 160 | 11.2 | 1252 | 12.5 | 0.333 | 25 |
| 39.447 | 0.22824 | (1 | 1 | 3 | 13 | 218 | 15.3 | 1654 | 16.5 | 0.322 | 26 |
| 43.203 | 0.20923 | (2 | 0 | 2) | 9 | 213 | 14.9 | 1591 | 15.9 | 0.317 | 27 |
| 47.549 | 0.19107 | (0 | 1 | 8) | 13 | 264 | 18.5 | 2584 | 25.8 | 0.416 | 20 |
| 48.55 | 0.18736 | (1 | 1 | 6) | 11 | 250 | 17.5 | 2322 | 23.2 | 0.395 | 22 |
| 56.555 | 0.16259 | (1 | 2 | 1) | 6 | 42 | 2.9 | 304 | 3 | 0.308 | 29 |
| 57.456 | 0.16026 | (1 | 2 | 2) | 13 | 97 | 6.8 | 819 | 8.2 | 0.359 | 25 |

2 theta: the diffracted angle, h,k,l: Miller indices of plane, BG: Back Ground, I: Intensity, Area: Area under the peak, FWHM: Full Width of Half Maximum, XS (nm): crystal size (nm).

**Table 5. PXRD analysis of nanopowder of (7) import chicken eggshells.**

| 2-Theta | d(nm) | (h | k | l) | BG | Height | I% | Area | I% | FWHM | XS(nm) |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 23.038 | 0.38574 | (0 | 1 | 2) | 8 | 141 | 8.9 | 692 | 9.2 | 0.283 | 28 |
| 29.357 | 0.30398 | (1 | 0 | 4) | 24 | 1533 | 100 | 10274 | 100 | 0.284 | 29 |
| 31.436 | 0.28434 | (0 | 0 | 6) | 15 | 39 | 2.2 | 147 | 2.5 | 0.223 | 37 |
| 35.946 | 0.24963 | (1 | 1 | 0) | 12 | 206 | 14.7 | 1077 | 13.4 | 0.283 | 29 |
| 39.399 | 0.22851 | (1 | 1 | 3) | 244 | 259 | 20.2 | 1897 | 16.9 | 0.33 | 25 |
| 43.195 | 0.20927 | (2 | 0 | 2) | 10 | 213 | 16.1 | 1492 | 13.9 | 0.29 | 29 |
| 47.501 | 0.19125 | (0 | 1 | 8) | 13 | 240 | 21.7 | 2640 | 15.7 | 0.423 | 20 |
| 48.543 | 0.18739 | (1 | 1 | 6) | 11 | 259 | 23.2 | 2304 | 16.9 | 0.4 | 21 |
| 56.598 | 0.16248 | (1 | 2 | 1) | 4 | 44 | 4.2 | 318 | 2.9 | 0.375 | 24 |
| 57.489 | 0.16017 | (1 | 2 | 2) | 8 | 91 | 11.8 | 873 | 5.9 | 0.422 | 21 |
| 58.141 | 0.15853 | (1 | 0 | 0) | 6 | 24 | 1.2 | 151 | 1.6 | 0.279 | 32 |

2 theta: the diffracted angle, h,k,l: Miller indices of plane, BG: Back Ground, I: Intensity, Area: Area under the peak, FWHM: Full Width of Half Maximum, XS (nm): crystal size (nm).

## Discussion:

Conferring to (30) shell is about 11.36% of the total weight of the local egg (local egg weight ~ 49 gram) and 9.69% of the total weight of the import egg (import egg weight ~ 68 gram) and it presents substances of calcium carbonate. So, egg shell is a rich basis of mineral salts, chiefly calcium carbonate type calcite form .

The eggshells contain chiefly $CaCO_3$, therefore calcium shows an essential part in the eggshells construction through FTIR and PXRD analysis. The previous studies showed that the chief structure material of the shell is $CaCO_3$ (96%), and the residual modules are magnesium, phosphorus, but also copper, zinc, iron and numerous trace elements, amongst them lithium, strontium and bar. They described that wholly egg shells obligated parallel biochemical substances that mostly consist of calcium carbonate and other additional elements; for example S, Mg, P, Al, K and Sr [31, 32, 33, 34].

These results confirm the studies by Solomon [35] and Dennis et al. [36] demonstrating calcite as the crystal construction of the Leghorn hen eggshells. A comparable outcome was described from the analysis of the biochemical structure of normal and boiled hen egg shells by XRD [37].

The eggshells ultrastructure was detected using SEM presented in (Fig. 1), obtained from raw local and import eggs. Under low and high magnification (20000-50000X), there was not much change in SEM micrograph between local and import chicken eggshells.

Both types of chicken eggshells express a particle size of ≤50 nm ($CaCO_3$-calcite form). The reduction of the crystal size can be recognized to the roll mall process. Also, the higher strength peaks for local and import chicken eggshells in FTIR and PXRD results could be associated with the decrease in the crystallite size. The XRD design peaks accorded the peaks of calcite CaCO3 ICDD No. 85-11108 marvelously and agree with the results of Kamkum et al. [3] and Murakami et al. [18].

SEM and TEM description is used to conclude the morphology of any kind of nanoparticles [38, 39, 40], especially here for $CaCO_3$ nano powders. The SEM images of the $CaCO_3$ from both types of chicken eggshells are presented in (Fig. 1 and 2). These images indicate that the $CaCO_3$ powders are between rhombohedral and spherical shape with distinguished pores distribution in the surface with a typical size of ~50 nm. The eggshell powder designates the existence of agglomeration. This is so because of the existence of other phosphate compounds in the formula of tricalcium phosphate (TCP). This remark agrees with the results described by Kalita and Verma [41] and Syafaat [42].

The chicken eggs are spent often in plenty amounts, but till now the shells are unused and static infrequently used particularly as a source of calcium or $CaCO_3$ material that can be good micro or nano-materials for the removal of toxic heavy metals as a noble substantial in the management of strong acidic waste water as well as a good neutralization capacity, development of a novel opportunity of pharmaceutical excipient, bone scaffold development, teeth manufacturing and filling; and drug carrier and delivery for bone disorders and diseases.

## Conclusion:

Chicken eggshells from the hatcheries, fast food productions and homes must not be predisposed in a method that contaminates the environment. The properties of local and import chicken eggshells are recognized with SEM, TEM, XRD and FTIR analyses. After analysis, the greatest structure of eggshell is ($CaCO_3$) in addition to the distributions of pores on the surface of nano-particles detected. From the results of this study, we recommend using chicken eggshell nano powder as a worthy substantial in the handling of tough acidic waste water over a reasonable application of heavy metal ions in addition a worthy deactivation capability, development of a novel opportunity of pharmaceutical excipient, bone scaffold development, teeth manufacturing and filling; and drug carrier and delivery for bone disorders and diseases.

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## Authors' declaration:

- Conflicts of Interest: None.
- We hereby confirm that all the Figures and Tables in the manuscript are mine ours. Besides, the Figures and images, which are not mine ours, have been given the permission for re-publication attached with the manuscript.
- The author has signed an animal welfare statement.
- Ethical Clearance: The project was approved by the local ethical committee in University of Mosul.

## Authors' contributions:

S.M., G.S., S.E. and A.A. contributed to the design and implementation of the research, to the analysis of the results and to the writing of the manuscript.

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# إعادة تدوير قشر بيض الدجاج إلى مسحوق نانوي: التركيب والخصائص

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**الخلاصة:**

تم تحسين الموارد غير التقليدية من الكالسيوم ($Ca^{+2}$) للطيور والأحياء المائية والحيوانات المحلية. تم التخطيط لهذا البحث لتحديد أكثر أنواع كربونات الكالسيوم تعددًا ($CaCO_3$) التي تحدث في نوعي قشر بيض الدجاج (النوع المحلي والنوع المستورد). في هذا البحث ، تمت العمل على التحليل المقارن لمحتوى كربونات الكالسيوم( $CaCO_3$) لقشر البيض من السلالة المحلية والمستوردة عبر اجراء تحليل المجهر الإلكتروني لمسح الانبعاث الميداني (FESEM) ، ومجهر الإرسال الإلكتروني (TEM) ، والتحليل الطيفي للأشعة تحت الحمراء (FTIR) و تحليل حيود الشعاع للمسحوق (PXRD). أظهرت النتائج أن قشر بيض الدجاج الأصلي والمستورد يشتمل على شكل الكالسيت الذي له شكل بين المعيني والكروي مع توزيع مسام مميز في السطح وحجم تبلور (31) نانومتر لقشر بيض الدجاج المحلي و (32) نانومتر لقشر بيض الدجاج المستورد على التوالي. يوجز الباحثون نتائجهم بأن قشر بيض الدجاج المحلي والمستورد يحتوي على أعلى موارد كربونات الكالسيوم ($CaCO_3$).

**الكلمات المفتاحية:** إعادة الاستخدام ، الإنتاج ، مسحوق النانو ،المجهر الاليكتروني،.التحليل الفيزيوكيميائي ، قشر بيض الدجاج .