*Research Article*

# Natural preparation of rice husk-derived silica and eggshell-derived calcium carbonate composite as a coating material for dental implant

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**Abstract:** Background: The world is in front of two emerging problems being scarceness of virgin resources for bioactive materials and the gathering of waste production. Employment of the surplus waste in the mainstream production can resolve these problems. The current study aimed to prepare and characterize a natural composite CaO-SiO2 based bioactive material derived from naturally sustained raw materials. Then deposit this innovative novel bioactive coating composite materials overlying Yttria-stabilized tetragonal zirconia substrate. Materials and method; Hen eggshell-derived calcium carbonate and rice husk-derived silica were extracted from natural resources to prepare the composite coating material. The manufactured powder was characterized via Fourier-transform infrared spectroscopy (FTIR), field emission scanning electron microscope (FESEM), X-ray fluorescence (XRF), X-ray diffraction (XRD) and particle size analyzer. The bioactive composite was deposited through radiofrequency (Rf) reactive magnetron sputtering overlying disc-shaped samples with a dimension of 10 mm diameter were prepared from partially sintered Yttria-stabilized tetragonal zirconia polycrystal (Y-TZP). Results: The particle size of the rice husk-derived ranged between (480.4 – 606.1) nm with a mean particle diameter of 541 nm. The eggshell derived calcium carbonate powder presented a particle size between (266.4-336) nm and a mean particle diameter of 299.9 nm. The XRD data revealed the crystalline nature and phase composition of the natural prepared calcium carbonate powder and demonstrate the monocrystalline nature of natural SiO2. FTIR spectrometer showed the emergence of novel spectra separated from the two innovative components. XRF analysis revealed that 99.4% of the rice husk is SiO2 while eggshell-derived powder is mainly composed of calcium oxide. Fe-SEM images of the coated zirconia exhibited average thickness of the natural CaCO3/SiO2 coat layer may reach to12.84 μ. Conclusion: The prepared composite derived from natural resource waste is suitable to be utilized as a coating material for ceramic dental implants with promising biological and mechanical properties.

**Keywords:** Rice husk; Silica; Eggshell; Calcium carbonate; Zirconia; Coating material.

## Introduction

Dental implants have been extensively utilized as a supporter for prosthodontic restorations; removable and fixed as well as the maxillofacial restorations, with a high degree of success (1). Different types of material were used as an implant; the most common is titanium and its alloy, later zirconia acquired robust interest due to many desirable properties (2). The aforementioned materials; titanium and zirconia regarded as a bioinert biomaterials which necessitate the use of bioactive biomaterial as a coating overlying the bioinert substrate; titanium and zirconia (3). The selection of the bioactive coat depends on physicochemical characteristics in addition to the availability and affordability of the material (4). In recent years, natural biocomposites received widespread attention as an active coating covering metallic implants; due to its bioactivity, availability and affordability (4). Micro-nano organizational modification of the surface of the implant may improve bone conductivity and hydrophilicity in addition to decreasing the conducted stress [5].

Calcium oxide/ Silicon dioxide built bioceramics have been considered as a probable alternative for artificial bone owing to their excellent biocompatibility and Osseoinductivity [6].

Among these biomaterials, autogenous bone is often Employment of by-products or leftover of agricultural operations received a wide consideration in the emerging technologies, scientific pursues and biological scopes in recent years [7].

Rice husk is an agricultural residue material plentifully available in Iraq and all rice-producing countries. Rice husk ash is rich in silica and can be used as a source for the manufacturing of silica powder [8]. Various authors proved that the rice husk ash was an exceptional resource for amorphous silica [9, 10].

Chicken eggshell agricultural junks represent the environmental pollution problem. The chemical composition of eggshells is composed mainly of calcium carbonate $CaCO_3$ [10].

Eggshell is regarded as massive pollution for the environment, at the same time rich source for $CaCO_3$ and CaO, making the opportunity to utilize eggshell as an alternative sustainable source for bioactive osseoconductive material [11].

The current study aims to prepare and characterize of a novel naturally prepared calcium carbonate ($CaCO_3$)/ silica $SiO_2$ composite based bioactive implant-coating material.

## Materials and Methods

### Preparation of biological silica

Subsequent to the milling process, the rice husk was sieved by using stainless steel mesh No. 230 and thoroughly washed with deionized water with the aid of mechanical stirring for 1 h then dehydrated by dry heat oven at 70°C. The rice husk was treated with a leaching agent (1 M hydrochloric acid) for 2 h at 90°C to minimize metallic contamination and rinsed with distilled water until reach neutral pH. The achieved mixture was then positioned in a furnace (VITA ZYRCOMAT 6000 MS, Germany) at 700°C for 2 h in order to accomplish the calcination process. Calcinated rice husk ash around 1000 mg was added into 20 mL of 1.5 M sodium hydroxide in a glass beaker for 1 h at 90°C to produce sodium silicate solution [12]. Sodium silicate was then dissolved in pure ethanol 250 Ml, then diluted with 1 L of distilled water for 10 min. The resultant solution was softly titrated with 3 M orthophosphoric acid until the formation of yellowish gel at neutral pH. This gel was washed with warm distilled water in order to remove any remnants of sodium silicate or sodium phosphate followed by centrifugation at speed of 4000 r/min for 15 min. The manufactured gel was then dehydrated at 90 °C for 2 h and calcinated in a furnace (VITA ZYRCOMAT 6000 MS, Germany) at 550 °C for 30 min to produce silica powder [13].

### Preparation of eggshell-derived $CaCO_3$ powder

Fresh chicken eggshells with accompanied internal membranes were firstly crushed in a mortar and pestle to a suitable particle size powder. Powder of eggshell with quantity around 100 grams was filtered by sieve No. 35. A ball milling machine was used for milling of the filtered powder with water producing slurry mixture, which was desiccated for 24 h at 105°C to attain fine powder. The dried powder was filtered using No. 230 sieve. Quantity powder of 10 grams was soaked in 50% bleaching agent (sodium hypochlorite) for 10 minutes. Followed by rinsing the powder with deionized water for 5 times in order to eliminate any bleach deposits. The powder was placed in a hot oven at 105°C for 24 h for drying. The dried powder was more ground by the means of an electrical grinding machine and filtered with No. 230 ASTM sieve to achieve $CaCO_3$ [14].

**Characterization of prepared powders**

The prepared powders; Rice husk-derived Silica and Eggshell-derived Calcium Carbonate were characterized with Fourier-transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), X-ray fluorescence (XRF) and particle size analyzer. After the characterization of the silica and calcium carbonate, the two powders were mixed with a mechanical stirrer at an ambient temperature according to the intended ratios (90% $CaCO_3$ with 10% $SiO_2$). The resultant mixture was investigated by powder particle size analyzer X-ray diffraction (XRD), Fourier-transform infrared spectroscopy (FTIR) and X-ray fluorescence (XRF) in order to determine the composition and concentration of elements

**Preparation of the specimens**

Disc-shaped samples with a dimension of 10 mm. diameter and 2 mm. thickness were prepared from partially sintered Yttria-stabilized tetragonal zirconia polycrystal (Y-TZP) Vita YZHT substrate by the means of Exocad dental computer aid design/computer aid manufacture (CAD/CAM), Imes Icore CORiTEC 250i [12]. The specimens were sintered with Vita ZYRCOMAT 6000 MS sintering furnace, 30 specimens with 10 mm diameter (10 specimens for each test) were recruited for x-ray diffraction (XRD) (Lab X, XRD 6000, SHIMADZU, Japan), field emission scanning electron microscope (FESEM) (Inspect f50 FE-SEM; Netherland), and x-ray fluorescence (XRF) (PAN analytical laboratories, Tehran, Iran).

**Coating procedure**

Weight of 20g of mixture calcium carbonate/silica (90/10 % W) powder was pressed in cylindrical stainless-steel mold using applied force around 30 Kg. with (dimension of 51mm diameter and 7mm height. Pressing is mechanical process used to reduce the porosity and vacancies between the particles and to produce a disc at 5cm diameter and

a 4mm thickness, then the discs were sintered at 900 °C in order to reach sufficient toughness to resist fracture during sputtering (17).

The bioactive composite was deposited through radiofrequency (Rf) reactive magnetron sputtering utilizing $SiO_2/CaCO_3$ as a sputtering target which have a (50 mm) diameter and (4 mm) thickness. The composition of reactive gas is composed of argon as sputtering gas. The base pressure in the vacuum chamber is $1\times10^{-5}$ Torr and the working pressure has been $6\times10^{-3}$. The distance between the target and substrate was 10 cm and the time of deposition was 20 h at 150°C temperature and at frequency equal to 13.56 MHz (18).

**Physical tests and structural characterization**

Field emission scanning electron microscope (FE-SEM) is an important microstructural analysis technique used for observing the characteristics of the compounds (19). In the present work, FE-SEM (Inspect f50 FE-SEM; Netherland) with an accelerating voltage of 10–20 Kv was used to reveal the microstructure of the experimental biological coat include naturally prepared rice husk-derived silica and eggshell-derived calcium carbonate composite. FE-SEM was used to diagnose the phases, distribution of particles as well as to characterize the morphology of the prepared specimens (20). X-ray fluorescence (XRF) data was analyzed to characterize the elemental composition of the coated zirconia substrate (22, 23). The

XRF analysis of the coating composition was accomplished at Arya electron optic LTD for advanced scientific and industrial equipment, North Shiraz Ave, Tehran, Iran. X-ray diffraction (Lab X, XRD 6000, SHIMADZU, Japan) had been used to inspect the coated specimens to examine the crystallographic orientation of the coating layer.

## Results

The particles size of the manufactured powders was examined by means of laser particle size analyzer. The particle size of the rice husk-derived silica shown in **Fig. 1(A)** ranged between (480.4 – 606.1) nm with mean particle diameter of 541 nm as. The eggshell derived calcium carbonate powder presented a particle size between (266.4 - 336) nm and a mean particle diameter of 299.9 nm **(Fig. 1(B)).**

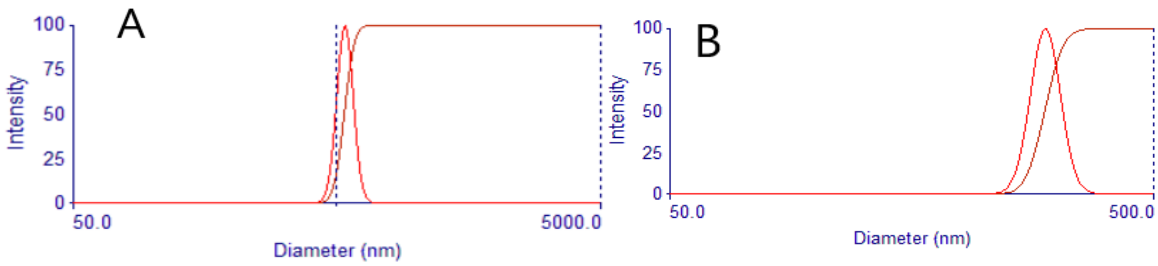


**Figure 1**: Size distribution of (A) Rh-sil ica; (B) Eggshell-Ca cium Carbonate

The X-ray diffraction pattern of the calcium carbonate reveals sharp and well-defined peaks at 2$\theta$ values of 23.2º, 24.9º, 36.1º, 39.6º, 43.2º, 47.6º and 48.6º. However, peaks are also perceived at 2$\theta$ values of 31.6º, 57.8º, 61.5º, 65.4º and 73.3º. The XRD pattern in **Fig.2** demonstrates the monocrystalline nature of natural $SiO_2$ which contests with reference No. (01-076-0941). An amorphous peak was recorded at 23º which is in agreement with a study conducted by Martinez *et al.* in 2006 [26].

**Figure 2:** XRD of the three powders

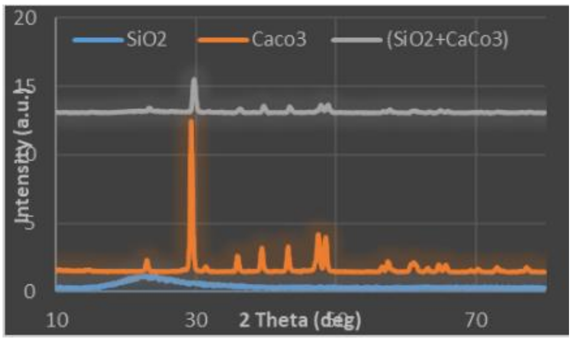


**Fig. 2** Revealed the diffractograms of the three powders; silica, calcium carbonate and composite (mixed extracted powders). The diffractogram of the composite material exhibits compromised spectra between the silica and calcium carbonate powders. **Fig. 3(A)** illustrate FTIR spectra of the rice husk-derived silica. The band about ~ 806 $cm^{-1}$ corresponds to Si-O bending vibration [26]. While **Fig. 3(B)** shows the FTIR spectra of the eggshell-derived calcium carbonate particles. The samples display an extensive absorption peak of $CO_3$ ions at ~1795 $cm^{-1}$, ~1458 $cm^{-1}$ ~1084 $cm^{-1}$, ~854 $cm^{-1}$, and~713 $cm^{-1}$ which have been itemized to be the common demonstrative features and the crucial styles of vibration of the carbonate ions present in calcium carbonate [25,27].

The composite powder spectrometer was established the emergence of novel spectra separated from the two innovative components ($CaCO_3$ and $SiO_2$) as presented in **Fig. 3(C)**.

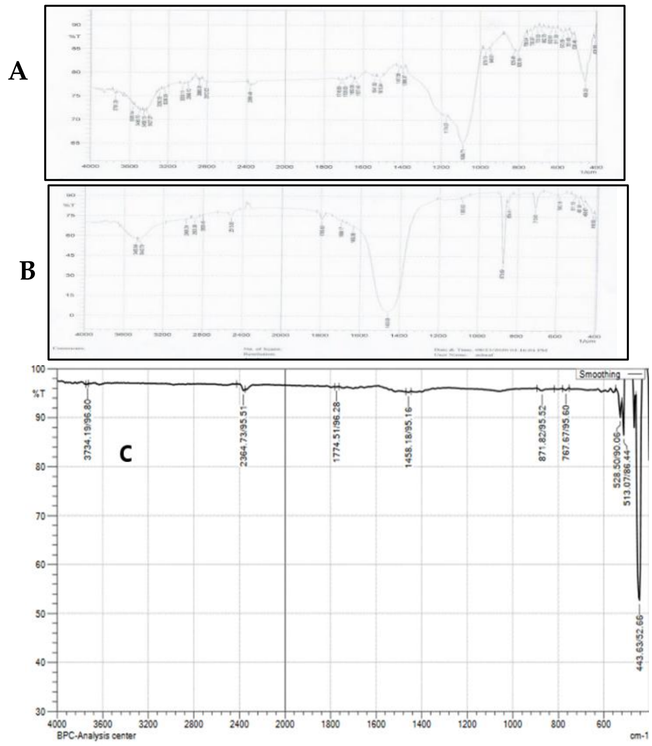


**Figure 3:** FTIR analysis of the
(A) Rice husk-derived silica;
(B) Eggshell-derived calcium carbonate; and
(C) The composite CaCO3/SiO2

Table 1 exhibits the chemical composition of the rice husk-derived powder by means of XRF analysis. Results revealed that 99.4% of the prepared powder is SiO2 in addition to small amounts of calcium oxide and ferric ions [28]. The XRF data of eggshell-derived powder revealed that the main composition is calcium oxide. The composite powder XRD proved that it consists of 90.2% calcium carbonate derived from CaCO3 with 9.7% of silica.

**Table 1.** Elemental analysis of the Rh-derived, eggshell- derived and composite powders

| | **Elements concentration (wt.%)** | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| **Prepared powder** | **$SiO_2$** | **CaO** | **MgO** | **P2O5** | **S** | **K** | **Fe** | **Na** |
| **Rh-derived** | 99.4 | 0.59 | - | - | T | - | T | - |
| **Eggshell- derived** | - | 98.2 | 1.33 | 0.45 | T | T | T | T |
| **Composite** | 9.7 | 88.2 | 1.0 | T* | T | - | - | T |

The finding of XRF analysis of coated specimen was demonstrated in Table 2. The results indicated a great quantity of CaO which referred to the presence of CaCO3, which is the main component of the composite coat (22). The cross-section fe-SEM images of the coated zirconia exhibited average thickness of the natural CaCO3/SiO2 coat layer may reach to12.84 μ as shown in Fig. 4(C).

**Table 2:** XRF Elemental analysis of coated zirconia substrate:

| Element | wt % | Element | Ppm |
|---|---|---|---|
| **$SiO_2$** | 4.255 | S | 1276 |
| **$Al_2O_3$** | 0.198 | Cl | 509 |
| **$Fe_2O_3$** | N | Ba | 32 |
| **CaO** | 45.5 | Co | N |
| **$Na_2O$** | 0.121 | Cr | N |
| **K2O** | N | Cu | 17 |
| **MgO** | 0.213 | Mo | N |
| **MnO** | 0.013 | Nb | 115 |
| **$TiO_2$** | 0.018 | Ni | 14 |
| **$P_2O_5$** | >40% | Pb | 107 |
| **LOI** | 3.18 | Rb | 46 |
| **$SO_3$** | 3.0972 | Sr | 116 |

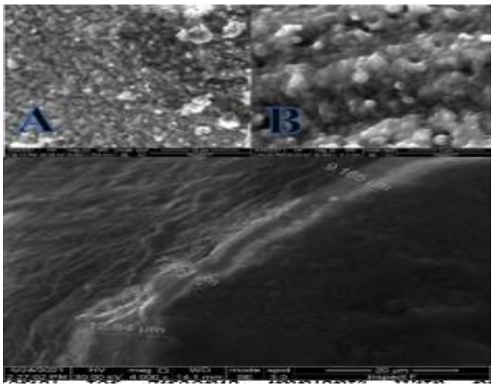


**Figure 4:** FeSEM images of surface topography of A) Uncoated zirconia; B) Natural CaCO3/SiO2. And C) FESEM cross-section images of natural CaCO3/SiO2 coat layer

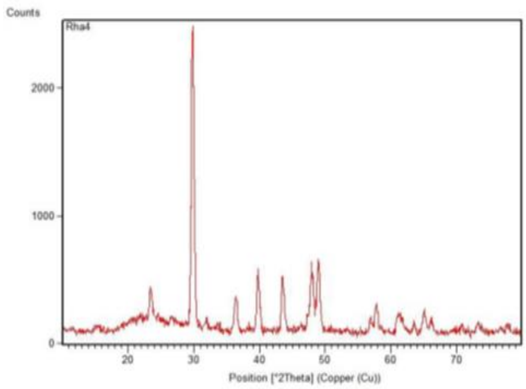


**Figure 5:** X-ray diffraction spectra of coated zirconia

**Discussion**

The XRD data revealed the crystalline nature and phase composition of the natural prepared calcium carbonate powder. The sharp peaks of the diffractogram indicating high crystallinity of the prepared powder [(25)]. The XRD analysis of the coated zirconia substrate illustrated in **Fig. 5.** The data acquired from the XRD pattern is identical with the diffractogram of the natural rice husk-derived silica/eggshell-derived $CaCO_3$ composite powder. FTIR spectra exhibited that the band neighboring 1089 $cm^{-1}$ indicates Si-O-Si lopsided stretching vibration when the connecting oxygen atom transfers corresponding to the Si-Si lines directed contrary to their Si adjacent lines [(27)]. While FTIR spectra of eggshell powder demonstrate features and the crucial styles of vibration of the carbonate ions present in calcium carbonate [(25,27)]. The composite powder spectrometer was established the emergence of novel spectra separated from the two innovative components. XRF data explore the excessive amount of calcium oxide indicating the high percentage of calcium carbonate, which is the main constituent of the eggshell [(29)]. The increased thickness of natural composite may be attributed to the high cohesion bonding between its particles (10). The coating surface microstructure of natural $CaCO_3/SiO_2$ on zirconia substrate appear porous as illustrated in the feSEM images in **Fig. 4 A&B.** These porosities may have a vital role for enhancing bone regeneration as well as increasing the surface area of the implant surface leading to decreasing the generated stress inside the contiguous bone (3).

## Conclusion

Recently, byproduct and waste management for the development of new products has gained immense interest. Within the limitations of the current study, a biological $CaCO_3/SiO_2$ composite was prepared from avian eggshell and rice husk via simple methods to be used as a coating material to the zirconia substrate. Therefore, it can be utilized effectively as a coating material for zirconia implants with predictable promising biological and mechanical properties.

**Conflict of interest:** None.

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**العنوان: تحضير الخليط الطبيعي من مادتي السيليكا المشتقة من قشور الرز, وكاربونات الكالسيوم المشتقة من قشور البيض كطلاء للزرعات السنيه**

**الباحثون: رحاب عامر كريم ,غسان عبد الحميد ناجي**

**المستخلص:**


الخلفية: مشكلتان عالميتان تلوح في الافق ,الاولى تتمثل بشحة مصادر المواد البايولوجية ذات الاستخدام الطبي الامن. اما الثانية تراكم المخلفات الطبيعية ذات الاضرار البيئية مثال على ذلك تراكم قشور الشلب الغير صالحة للعلف الحيواني مسبباً تلوف بيئي عند احتراقها نتيجة تولد غازات ثقيلة. مثال اخر يتمثل بقشور البيض مصدر تلوث بيئي كبير وصعوبة التخلص منها.

مواد وطرق العمل: ان هذه الدراسة تهدف لاستخلاص السيليكا وكاربونات الكالسيوم من قشور الشلب وقشور البيض على التوالي. وتحضير خليط منهما لعمل مادة طلائية محفزة لعملية تجديد العظم حول الزرعة لتفادي نقص العظم المحيط بالزرعة وفشلها. ومن ثم بعد تحضير المادة واجراء الفحوصات اللازمة يتم استخدامها لاكساء زرعات الزكونيا بطريقة الترذيذ بولسطة المعجل المغنطي. تم خلال هذه الدراسة الاستعانة بالفحوصات التالية: اشعة فيورير تحت الحمراء, الماسح الالكتروني, فلورة الاشعة السينية , و مشتت الاشعة السينية. تم فحص عشرة عينات لكل فحص من الفحوصات المذكورة آنفاً. كل عينة بشكل قرص بقطر ١٠ ملم. كذلك تم قياس حجم ذرات كل من المسحوقين المستخلصين.

الاستنتاجات: ان هذه المواد المستخلصة من المخلفات الطبيعية يمكن الاستفادة منها لاكساء غرسات الاسنان المصنوعة من مادة الزركونيا بنتائج واعدة