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Bio and Mechanical Evaluation of an Enhanced Bio Glass

Abstract- *These Tap casting and powder metallurgy methods used to produce the 45S5 Bioglass. This study revealed that the bioglass modified with 0.2% Y_2O_3 has about 700% increases in hardness. The modification of bioglass with 0.2% Y_2O_3 leads to a 44% increase in fracture toughness values. This study revealed that the 0.2% Y_2O_3 bioglass composition-modification improves the fracture strength by almost 150%. The laboratory histological sections showed that 45S5 bioglass (original composition materials showed nether systemic nor local inflammation with new bone formation at site of implantation. The Y_2O_3 modified the 45S5 bioglass showed no inflammation reactions as well, but no new bone formation and regeneration was noticed in the adjacent bone tissue.*

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1. Introduction

Natural bone is a compound substance of collagen and calcium phosphate, which is the mineral component of the bone. The mineral component in the bone forms 60 to 70% of the total bone structure. This mineral component consists of the carbonate ion, which contains carbonates, as well as a small percentage of magnesium, fluorine and very few other elements. The crystalline structure of calcium phosphate in the natural bone is very much like the crystalline structure of hydroxyapatite [1,2].

For the material to be considered bioactive, it must show a biological response to the living tissue of the body. The great development of biomaterials has led to the development of many systems that can be used as alternatives to natural bone in the case of loss or fracture or bone diseases to the normal natural bone restoration itself.

Bio-glass is one of the systems developed and used in these applications. Hydroxyapatite (HA), B-tri calcium phosphate (b-TCP) and their composites are widely because of their high biocompatibility and osteointegration [3].

Bio-glass is a mixture of alumina, silica, magnesia, and a group of oxides for essential elements such as sodium, calcium and phosphorus.

Bio-glass consists of many families each of them consists of different compositions like Bioglass TM (45S5) which is used currently in bone grafting application [4-6]. Bioglass material is composed of several components that occur naturally in the body (SiO, Ca, NaO, H, and P), and the molecular proportions of the calcium and phosphorous oxides are like those in the bones [7].

45S5 bioglass consist from 46.1 mol% SiC, 26.9 mol% CaO, 24.4 Na_2O_3 mol%, and 2.6 mol% of P_2O_5. This type of bioglass can form HCAP meantime two hours and bind with living tissues [8].

This study aims to increase the mechanical properties of the original bioglass by adding more active ceramic materials putting in consolidation biocompatibility with living tissue. Accordingly, in this study, yttrium oxide was inserted to 45S5 bioglass utilizing tap casting and powder metallurgy methods. Fracture toughness was analyzed using indentation technique.

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Microstructure and biological response was analyzed using scanning electron microscope. An animal model used to study the biological response of proposed system when implanted within living tissue.

2. Experimental Part

I. Preparation of the modified 45S5 Bioglass

45S5 Bioglass was prepared the following steps:

1- Dry mixing of bioglass components was carried out of 45wt% SiO_2, 25wt% Na_2O, 25wt% CaO and 5wt%P_2O_5. While 0.2wt% of Y_2O_3 was added during mixing stage as modifier.

2- Melting process applied to the mixed components at a temperature of 1350°C for 90 min. after melting of the mixed components, the melted mixture was rapidly poured on a cleaned stainless-steel plate. Then, the glass was crashed to get powder with average particle size of about 65 µm.

3- Cold pressing method used to fabricate the testing samples using cylindrical die with a diameter of 7.5 mm. Instron tensile machine was used to form the powder within the die at a pressure of 290 MPa.

4- Finally, sintering process at 1100°C done to perform the final shaped samples for which were used in all tests.

II. Mechanical properties evaluation

Hardness of the bioglass samples was measured using Digital Micro-Vickers Hardness tester TH714) (Beijing TIME High Technology Ltd./China). Fracture toughness (KIC) was calculated using Vickers indentation method by measuring the initiated radial or median cracks around the Vickers indentation. SEM was used to monitor and measuring the dimensions of the crack (Figure 1).

Eq. (1) applied to determine the fracture toughness according to Ponton and Rawling work [9].

$$KIC=0.079\ (P/a3/2)\ \log\ (4.5\ a/C) \quad (1)$$

Where:
P: load (N)
a: Vickers indent half-diagonal (mm),
C: surface crack radius (mm) = a + L
L: length of crack (mm). 0.6 < C/a < 4.5

Diametrical compression test [10] used to perform the fracture strength for the bioglass samples using the following equation:

$$\sigma f = 2P / \pi dt \quad (2)$$

Where:
P: maximum load (N)
d: diameter of the specimen (mm) and
t: thickness of the test specimen (mm).

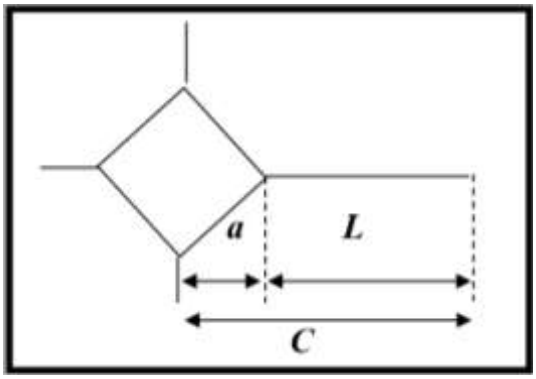

Figure 1. Crack measurement using Vickers indentation

II. Density and porosity evaluation

Archimedes method was used calculate density and porosity for the sintered samples. The samples were dry weighted first, then after socked in distilled water. The following equations was used to calculate the density and porosity [11]:

$$Bulk\ volume = \frac{Wsa - Wsw}{D} \quad (3)$$

$$Bulk\ density = \frac{W_D}{Wsa - Wsw} \times D \quad (3)$$

$$Apparent\ solid\ volume = \frac{W_D - Wsw}{D} \quad (5)$$

$$Apparent\ solid\ density = \frac{W_D}{W_D - Wsw} \times D \quad (6)$$

Where:
$\mathbf{W_{sa}}$ weight of a soaked sample and suspended in air,
$\mathbf{W_{sw}}$ weight of a soaked sample and suspended in room temperature distilled water,
$\mathbf{W_D}$ weight of a dry sample without soaking,
D density water.

The porosity can be determined depending on previous calculation and using the following equations [12]:

$$Apparent\ porosity\% = \left[1 - \frac{bulk\ density}{apparent\ solid\ density}\right] \times 100 \quad (7)$$

$$True\ porosity\% = \left[1 - \frac{bulk\ density}{true\ density}\right] \times 100 \quad (8)$$

II. Animal Model in vivo test

Vivo test was used to describe the biological reactivity of prepared bioglass with original composition compared to modified structure. Number and size of implants to be tested, have direct influence on the species of animal, which was chosen for a study. Some implant's designs are most commonly used in animal models like a screw type, or cylindrical (rod shaped). The implants with cylindrical shape are dependent on exact fit in order to be stable within implantation zone at the bone to give accurate results regarding their effect on bone integration. In this work, cylindrical shape implants were used.

Four healthy (4-6 months) old random bred SD rats weighing (300-400g) were used in this study. The rats were separates into two groups:

In-group (I), bone segment was crafted at the mid shaft of radius bone and left without any further treatment, and considered as a control group. In-group (II), a same bone segment was crafted and filled with bio composite material, which was prepared previously and considered as treated group.

All animals were injected with Acepromazine maleate (10 mg/kg BW.) I/M as a tranquillizer. After that, the animals were injected again with a mixture of ketamine hydrochloride (35 mg/kg BW.), and xylazine (5mg/kg BW.) I/M. During the surgery, the subcutaneous tissue was cut; after that, a blunt dissection between the pronator terse muscle and flexor carpi radials muscle was further made. The bone segment was cut by using an electrical saw, and then washed with normal saline. The segmental defect filled by bioglass sample in the treated group, and in control group the defect (fracture zone) left without additives. After 60 days, the histological slides were performed, Rats were killed, and the femur was exposed.

Bones were processed for light –microscope histology to establish their histogenesis, by using the histokinate. This processing involves the following steps:

1- Dehydration: is the removal of all extractable water by alcohol upgrading starting from 70%, through absolute alcohol (70%, 80%, 90%, and 100%), twice for 2 hours each time for each step to improve complete dehydration.

2- Clearing: As a dehydrator is removed, the tissue cleared, becoming translucent by using of xylene twice for 1.5 hour for each time.

3- Impregnation: Is the complete removal of xylene by substitution of paraffin penetration through the tissue used twice for 2 hours each time in paraffin bath adjusted on 58^{o}C.

4- Embedding: The processed tissue was oriented in melted paraffin, which provides affirm medium for keeping intact all parts of the bone tissue when sectioned.

5- Cutting: serial paraffin sections of 6-8 μm were cut by using rotary microtome.

6- staining of histological sections: routine histological stain of Harris haematoxylin and eosin stain.

3. Results and Discussion

I. Mechanical Properties

Image of SEM for 45S5 bioglass sample (Figure 2) shows an effective sintering process on the adhesion between particles. Where the 45S5 bioglass mixed with naphthalene pore-creator.

Vickers indentation image shown in Figure 3 which was adopted for hardness and fracture toughness measurements for both of original and Y_2O_3 - modified 45S5 bioglass. The effect of Y_2O_3 modifier on the mechanical properties of the 45S5 BIOGLASS system is shown in Table 1.

The enhancement in mechanical properties after the addition of Y_2O_3 may be take place due to the densification process for the contents after yttrium oxide addition to the matrix bioglass. The modifier has significant effect on the diffusion mechanism by inhibit boundary migration by solute drag mechanism [11,12]. The Y_2O_3 addition to the original composition bioglass showed an excellent enhancement, in fracture strength, over the original bioglass ceramic. Y_2O_3 seems to have the same effect when adding to MgO-CaO-SiO_2-P_2O_5-CaF_2 [13,14]. The effect of Y_2O_3 on the physical properties are listed in Table 2. The decreasing in the bulk volume led to increase the bulk density.

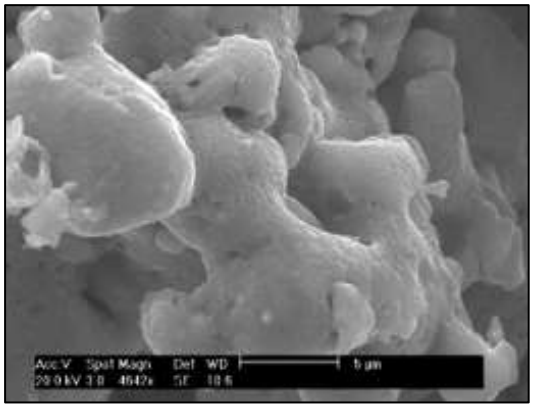

Figure 2: Micrograph of 45S5 bioglass mixed with naphthalene pore-creator

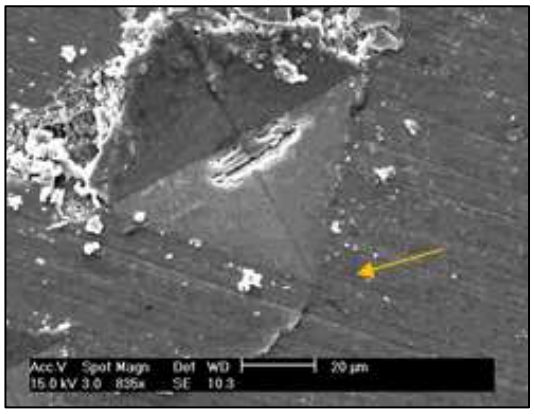

Figure 3: SEM micrograph showing initiated crack by Vickers indentation method*II. Physical Properties*

The modifier addition leads to an increase in the apparent of solid density, at the same time decreases the apparent density as presented in Table 2. A clear increase in apparent porosity and decrease in true porosity (Table 2) obtained duo to densification effect of the modifier oxide.

It is believed that Y_2O_3 reduce or attract the vacancies within the system and helps to push the others to the surface [13,15].

From results listed in Table 2, the densification effect is very noticeable on the physical properties due to Y_2O_3 addition.

III. Histological Evaluation

Histological sections (Figure 4) of the parent composite material was examined and showed that this material has good acceptance with the living tissue (SD rat), however, it also showed that this material can be intermixed with the original bone by diffusion of blood and extracellular fluid into the pores that are already present in the parent material.

Table 1*: Compressive strength, hardness and fracture toughness values for 45S5 bioglass*

Sample	**K_{IC}(MPa $\sqrt{m}$)**	**σ_t (MPa)**	**H_V**
45S5 bioglass	2.003±0.021	18.15 ±1.52	687 ±42
45S5 bioglass +Y_2O_3	2.888±0.123	42.6 ±4.12	5214 ±152

Table 2. Effect of modifier on the physical properties for the bioglass

Sample	**Bulk volume (cm^3)**	**Bulk density (g/cm^3)**	**Apparent volume (cm^3)**	**Apparent density (g/cm^3)**	**Apparent porosity (%)**	**True Porosity (%)**
45S5 bioglass original	0.441 ±0.14	2.264	0.119	3.0746	26.4±1.62	14.8±0.62
45S5 bioglass + Y2O3	0.421 ±0.18	2.375	0.110	3.3908	29.9±2.03	11.1±0.53

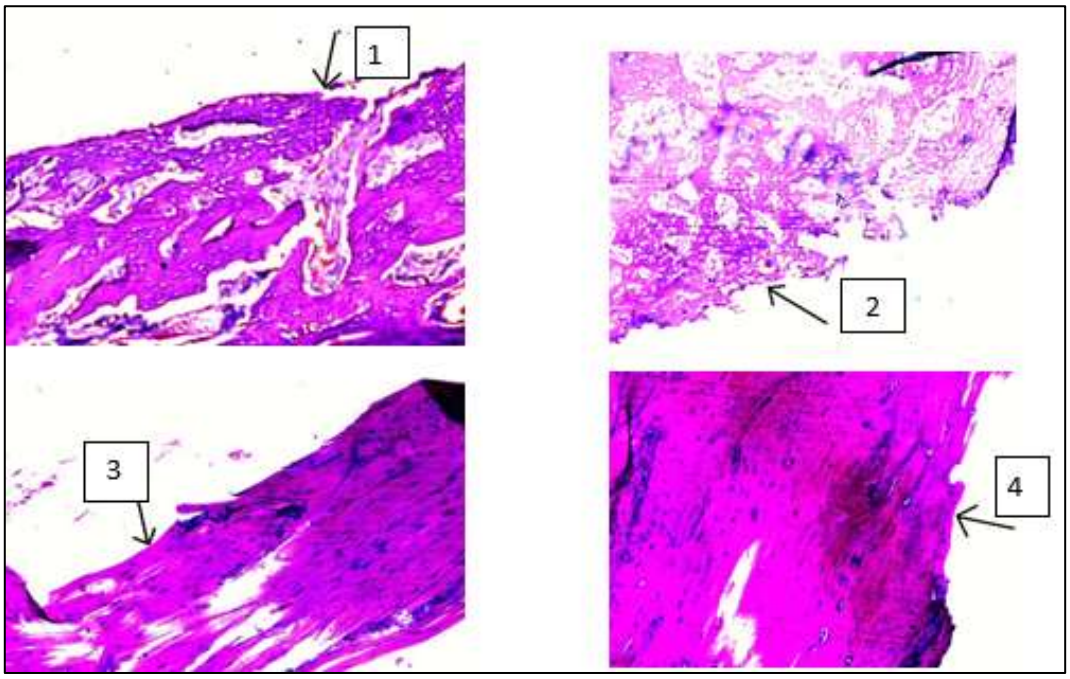

Figure 4. Histological evaluation for implants
(1) normal bony tissue,2 weeks (2) normal bony tissue 3 weeks
(3) normal periosteum 2 weeks for modified material (4) normal periosteum 3 weeks for modified material

Comparison was made with histological sections of the modified 45S5 Bioglass after adding the Y2O3 to it, showing that it will not decrease the biological compatibility in the living tissue neither systemic (fever, rash, rigor, tachycardia, sweeting and hypotension) nor local inflammation (redness, swelling, tenderness, increase local temperature, and loss of motion), while on the other hand it will not stimulate new bony formation (during the 3 week period), since the increase in the density of the Material due to modifier effect, the size of the pores decreased a little bit and this leads to more difficulty in passing of blood and extracellular fluid through it and this will have effect on decreasing regional new bone cells formation.

4. Conclusions

- A big enhancement in both of physical and mechanical properties were recorded after The Y_2O_3 addition due to its effect on densification and mechanism of diffusion by enhancing the concentration of point defect, mainly vacancies, and prevent the boundary migration.
- This study revealed that the bioglass modified with 0.2%Y_2O_3 has about 700% increases in hardness (i.e. from 687 to 5214 Hv).
- The modification of bioglass with 0.2%Y_2O_3 leads to a 44% increase in fracture toughness values (i.e. from 2.003 to 2.888 MPa/m).
- This study revealed that the 0.2% Y_2O_3 bioglass composition-modification improves the fracture strength by almost 150% (i.e. from 18.15 to 42.6 MPa).
- The 45S5 bioglass (original composition materials showed nether systemic nor local inflammation with new bone formation at site of implantation
- The Y_2O_3 modified the 45S5 bioglass presented no reactions of inflammation, and these is no new bone regeneration or formation was appeared in the adjacent bone tissue.
- Although Y_2O_3 addition decreased bioactivity of the original composition bioglass material but it was still accepted by the body since no inflammation were found.

More studies are recommended first for lone term reaction between the modified materials and living tissue, second for more researches about serious implantation of it in human being.

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