

Preparing and Studying the Effect of HA and ZrO₂ Addition on Fracture Strength of Dental Ceramic

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Abstract

This work aims at preparing and studying the effect of hydroxyapatite (HA) and zirconia (ZrO₂) additions on some physical properties and fracture strength of dental ceramic. In this work, dental ceramic batches have been prepared from (80% Potash feldspar, 15% silica and 5% Duekhla kaolin). Firstly, HA was added with different percentages (2,4,6,8,10)%wt. Secondly, both of HA and ZrO₂ have been added in percentage (5%wt) with different weight percentages for both of HA and ZrO₂ to (95%wt) of dental ceramic batch. Mixes were semi-dry pressed under (15 MPa) load, and they were fired at (1100°C) temperature. The results show the fracture strength of the dental that ceramic increases with the addition of HA. The best addition of HA was (4 %wt) because it gives the best of the physical and mechanical properties. By the addition of HA:ZrO₂ mixture, the value of fracture strength increased more than the addition of HA alone.

Keywords: Dental ceramic; Hydroxyapatite; Zirconia; fracture strength.

تحضير ودراسة تأثير إضافة الهيدروكسي اباتيت والزركونيا على مقاومة الكسر لسيراميك الأسنان

الخلاصة

يهدف هذا البحث إلى تحضير ودراسة تأثير إضافة الهيدروكسي اباتيت (HA) والزركونيا (ZrO₂) على بعض الخواص الفيزيائية ومقاومة الكسر لسيراميك الأسنان. في هذا العمل، تم تحضير خلطة سيراميك الأسنان مكونة من المواد التالية (80% فلدسبار البوتاسيوم، 15% سليكا و 5% كاؤولين دويخلة). تم أولاً إضافة مادة (HA) بنسب وزنيه مختلفة (2،4،6،8،10) % إلى سيراميك الأسنان. ثانياً، تم إضافة (HA) و (ZrO₂) معاً بنسبة وزنيه (5%) لكن بنسب إضافة مختلفة لكل من (HA) و (ZrO₂) إلى (95%wt) من خلطة سيراميك الأسنان. شكلت النماذج بطريقة الكبس شبة الجاف تحت ضغط (15 MPa) وحرقت بدرجة حرارة (1100°C). أوضحت النتائج إلى حصول زيادة في مقاومة الكسر لسيراميك الأسنان بإضافة الـ (HA) حيث كانت أفضل نسبة إضافة هي (4%HA) لأنها أعطت أفضل الخواص. بإضافة خليط من مادتي HA:ZrO₂ أدى إلى زيادة في مقاومة الكسر أكثر من إضافة مادة الـ (HA) لوحدها.

Introduction

Ceramics are inorganic nonmetallic mainly oxides and includes carbide, nitrides, borides ...etc. This class of materials include both traditional ceramics such as clay, porcelain, and glass, as well as modern technical

ceramics such as carbides, borides, oxides, and nitrides of various elements, which are used in high-technology applications [1,2].

Bioceramics

Bioceramics is a group of ceramics, which are biologically

active materials rich in calcium and phosphate [3]. Bioceramics is used in the human body as an artificial bone substitute in orthopedic and dental applications due to its biocompatibility. The response of these materials varies from nearly inert to bioactive to restorable. Nearly inert bioceramics include alumina (Al₂O₃) and zirconia (ZrO₂). Bioactive ceramics includes hydroxyapatite (Ca₁₀(PO₄)₆(OH)₂) and some special glass [1]. Hydroxyapatite and tricalcium phosphate (Ca₃(PO₄)₂) are similar in composition to bone and teeth and can be used for augmentation of alveolar ridges and filling bony defects [4].

Classification of Dental Ceramics

Dental ceramics can be classified in a variety of ways [3,4]:

1. Based on composition:
Silicate ceramics, main component is silica SiO₂. Oxide ceramics contains a principal crystalline phase like alumina. Oxide of zirconia has very high fracture toughness.
2. Based on type:
Feldspathic porcelain. Leucite – reinforced porcelain., Aluminous, porcelain. Glass infiltrated, alumina. Glass, infiltrated zirconia. and Glass ceramics.
3. Based on firing temperature:
Ultra–low fusing < 850°C, low fusing 850°C – 1093°C, medium fusing 1100°C – 1260°C, high fusing 1300°C.
4. Based on sub-structure metal:
Cast Metal, swaged metal, glass ceramics.

5. Based on use or indications:
Denture teeth fixed partial dentures, full crowns veneers, inlays post and cores.

Feldspathic Porcelain

Feldspars are used in the preparation of many dental types of porcelain. Potassium and sodium feldspar are naturally occurring minerals. The most important property of feldspar is its tendency to form glassy leucite when melted. Leucite is potassium aluminium silicate mineral with large co-efficient of thermal expansion compared with glasses [5].

Experimental Part

1. Raw Materials

The materials used for this work were:

- a. Potash feldspar. This material was brought from college of fine arts imported from the British Winger company.
- b. Silica (SiO₂). It was brought from Iraqi local markets as flint with purity of (99.8%).
- c. Duekhla kaolin (Al₂O₃.2SiO₂. 2H₂O). It was brought from Iraqi Geological Surveying Company.
- d. Hydroxyapatite (HA), It was prepared in laboratory.
- e. Zirconia (ZrO₂). It was brought from Iraqi local markets with purity of (99.98%).

2. Specimens Preparation

Kaolin rocks were grounded by using ceramic ball mill and then sieved to obtain kaolin powder in particle size (<53µm). While the other materials were received as fine powders. Dental ceramic batch was prepared from (80% Feldspar, 15% Silica and 5%

Kaolin). This batch was mixed by using ball mill for (1) hr.

Hydroxyapatite (HA) and zirconia (ZrO₂) were used as additive materials to dental ceramic batch to improve its fracture strength. The first group of samples includes, HA addition in different percentages (2,4,6,8 and 10) %wt. The second group, both of HA and ZrO₂ were added at the percentage of (5%wt) but with different weight percentages for both of HA and ZrO₂ (as revealed in table (1)) to (95%wt) of dental ceramic batch.

Uniaxial semi-dry pressing technique was employed for forming ceramic samples. The final powders for each group were mixed with (2%wt) poly vinyl alcohol (PVA) as a binder's material, and the batches were mixed for 30 min to insure homogeneity. The pressing of batch powders was done by a hydraulic pressing machine in a tool steel die of (2cm) in diameter with pressing pressure (15) MPa in a hydraulic pressing machine with a capacity of (40) MPa and then the samples were dried in an oven at (110°C) for (6) hrs. The sintering of samples was done on a brick base in a programmable furnace at a temperature of (1100)°C and soaking time (1) hr with a heating rate of (5°C/min) till it reached its required temperature.

3. Tests

3.1 Physical Tests of Samples:

The following tests were used to evaluate the physical properties of the samples [6]:

a. Linear shrinkage (L.Sh):

The linear shrinkage was measured depending on diametrical change using micrometer, before and after sintering.

$$(L.Sh)\% = \frac{d_0 - d}{d_0} \times 100$$

Where:

d₀: Sample diameter after pressing (mm).

d: Sample diameter after sintering (mm).

b. Bulk density (B.D):

The bulk density was measured using Archimedes method (depending on weighting of sintered samples) and the (B.D) values were obtained using the following formula:

$$(B.D) = \frac{W_d}{W_s - W_n} \times D$$

Where:

W_d: Weight of the dry sample after sintering (g).

W_n: Weight of the sample after immersing it in a distilled water and suspended in air through a balance (g).

W_s: Weight of the sample after immersing it in distilled water for 24hrs (g).

D: Density of distilled water (1 g/cm³).

c. Apparent porosity (A.P):

Apparent porosity was measured by the following equation:

$$(A.P)\% = \frac{W_s - W_d}{W_s - W_n} \times 100$$

3.2 Fracture Strength

Fracture Strength was carried out by using the diametrical compression disc test (Brazilian test) where the disc was placed between two surfaces and applied the load until fracture occurs [7,8], as shown in fig. (1), then the force obtained at fracture was recorded, then the following equation was applied to calculate the strength of material:

$$\sigma_f = \frac{2P}{\pi TD}$$

σ_f : fracture strength (N/mm²).

P: applied load (N).

D: sample diameter (mm).

T: sample thickness (mm).

4. Results and Discussion

4.1 Effect of (HA) Addition on Some Physical and Mechanical Properties:

Fig. (2) shows the linear shrinkage percentage variations with (HA) additions in dental ceramic batch. It can be shown that the linear shrinkage percentage decreases with the increase of (HA) addition in dental ceramic batch. This is due to low shrinkage of (HA), which is considered a non-plastic material which reduces the shrinkage in the ceramic specimens. The kaolin contents will reduce at the increase of the (HA) addition which represents the plastic material in the sample.

Fig. (3) shows the effect of the (HA) addition on the bulk density of dental ceramic. It seems that the value of the bulk density increases with the increase of (HA) content. The maximum bulk density was (2.42) g/cm³ at (10% HA) and the minimum bulk density was (2.25)

g/cm³ at (0% HA). The increase in density is due to the (HA) which has high density of (3.1)g/cm³ compared with the other materials that formed the dental ceramic.

Fig. (4) shows the effect of the (HA) addition on the apparent porosity of dental ceramic. It shows that the apparent porosity reduced from (8.4%) to (5%) at (4% HA) addition. This may be due to the formation of new secondary phases like (CaSiO₄, Ca₃(PO₄)₂ and α -Ca₂P₂O₇). These phases increase the bonding between the particles and fill the voids and pores. This leads to reduce the porosities in the samples [9]. When the (HA) content in dental ceramic increases more than (4%), the porosities will increase gradually. This may be attributed to arising the difference in coefficient of linear expansion of the new secondary phases and glassy phase. These may cause the crack that appears around the grain boundaries of the phases.

Fig. (5) shows the effect of the (HA) addition on the fracture strength of dental ceramic. It seems that the fracture strength increases with the increase of (HA) content in the dental ceramic batch. Fracture strength increased from (12) MPa to (17.95) MPa at the addition of 4% HA. This is due to porosity reduction which has a great effect on the mechanical properties of ceramic samples and also because of the formation of strengthening phases (Ca₂P₂O₇ and CaSiO₄) [9,10]. The value of fracture strength reduced with the increase of (HA) content at the

addition above 4%, because the increase of porosity.

4.2 Effect of (HA:ZrO₂) Addition on Some Physical and Mechanical Properties:

Fig. (6) shows the effect of the (HA:ZrO₂) additions on the bulk density of dental ceramic. It shows that the bulk density increases with increasing (ZrO₂) content from sample no. (0) to sample no. (5). This is due to the high density of the zirconia (6 g/cm³). The value of density has been reduced in sample no. (6) which has (5% ZrO₂ and 0% HA). This may be attributed to the effect of the fine powder of zirconia material which is about (5μm) filling the pores.

The effect of the (HA:ZrO₂) additions on the apparent porosity of dental ceramic was revealed in fig. (7). It observes that the content of zirconia is increased, the value of the apparent porosity is increased from the sample no. (1) to the sample no. (5). This is due to the melting point of the zirconia (2680 °C) which is higher than other materials and because of the reduced the glassy phase content, which create permeable open pores in the samples.

The value of fracture strength of the dental ceramic has been increased with the addition of HA:ZrO₂ mixture additions as shown in fig. (8). Sample no. (3) which contains (60% HA and 40% ZrO₂) has maximum fracture strength. This may be attributed to the formation of the strengthening phase (ZrSiO₄), which increases the bonding between the particles and formed phases [11]. Also the figure shows that the value of the

fracture strength of sample no. (3) has reduced and this may be due to the formation of cracks around zirconia particles, and because of the difference in coefficient of linear expansion of the strengthening phase (ZrSiO₄) and the glassy phase.

5. Conclusions

The main conclusions, which can be deduced from the present work, can be summarized as follows:

1. Fracture strength of the dental ceramic increases with the addition of HA. The best addition of HA was found to be (4 %wt), because it gives the best physical and mechanical properties.
2. At the addition of HA:ZrO₂ mixture, the value of fracture strength increases more than HA alone. The sample of 60%HA and 40%ZrO₂ has the maximum value of fracture strength (20.88 MPa).
3. Mechanical properties of dental ceramic and especially fracture strength decrease with the increase of the apparent porosity.

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Table (1) shows compositions of specimens that have (HA:ZrO₂) addition.

95 %wt	Dental ceramic batch							
5 %wt	HA %	0	100	80	60	40	20	0
	ZrO ₂ %	0	0	20	40	60	80	100
100%wt	Sample No.	0	1	2	3	4	5	6

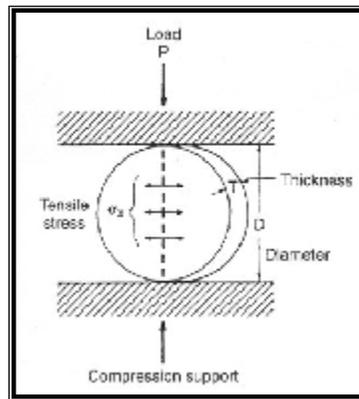


Figure (1) shows the specimen place during diametrical compression test [5].

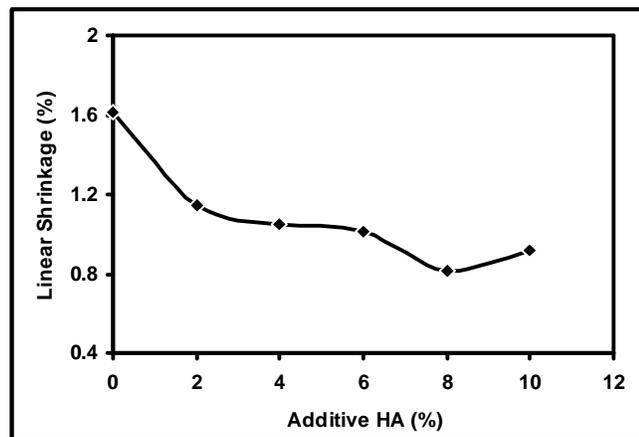


Figure (2) shows the effect of (HA) addition on linear shrinkage of sintered dental ceramic batch compact.

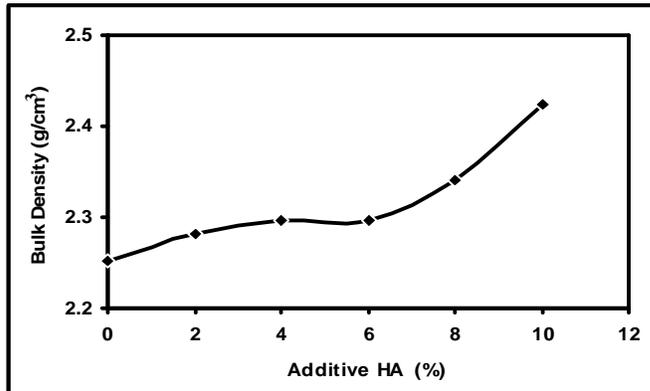


Figure (3) shows the effect of (HA) addition on bulk density of sintered dental ceramic batch compact.

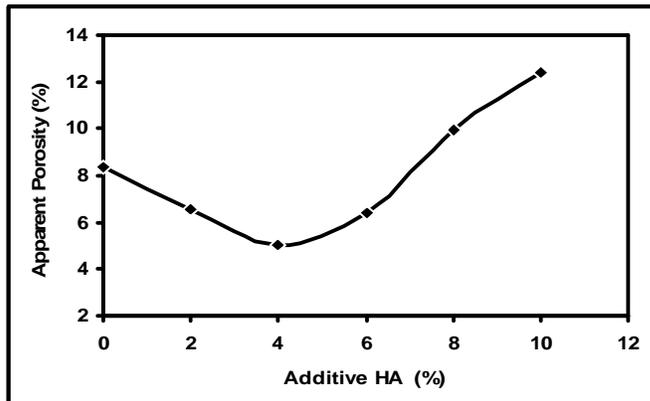


Figure (4) shows the effect of (HA) addition on apparent porosity of sintered dental ceramic batch compact.

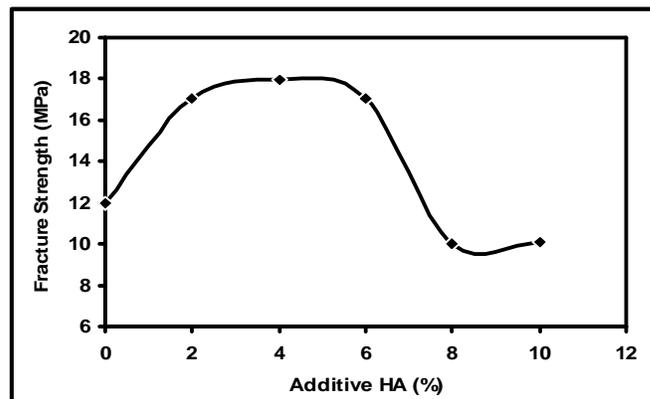


Figure (5) shows the effect of (HA) addition on fracture strength of sintered dental ceramic batch compact.

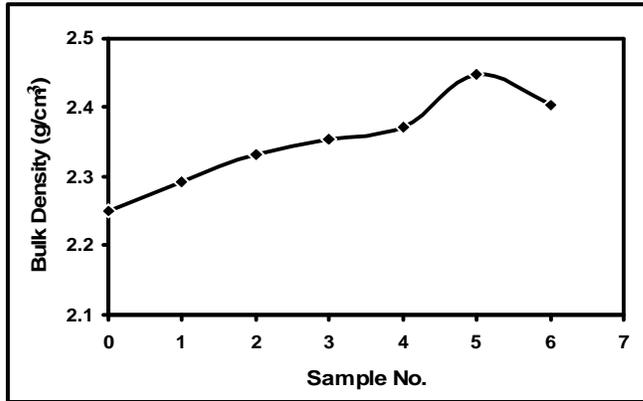


Figure (6) shows the effect of (HA:ZrO₂) addition on bulk density of sintered dental ceramic batch compact.

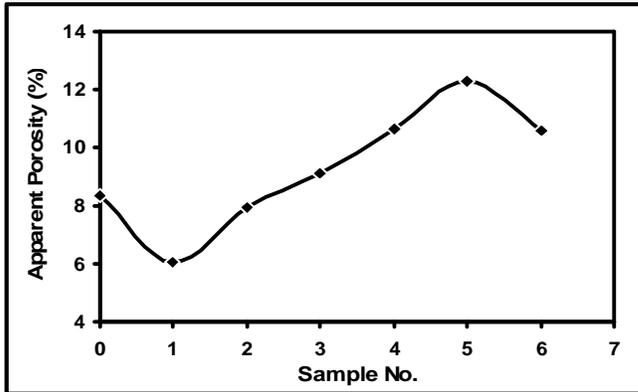


Figure (7) shows the effect of (HA:ZrO₂) addition on apparent porosity of sintered dental ceramic batch compact.

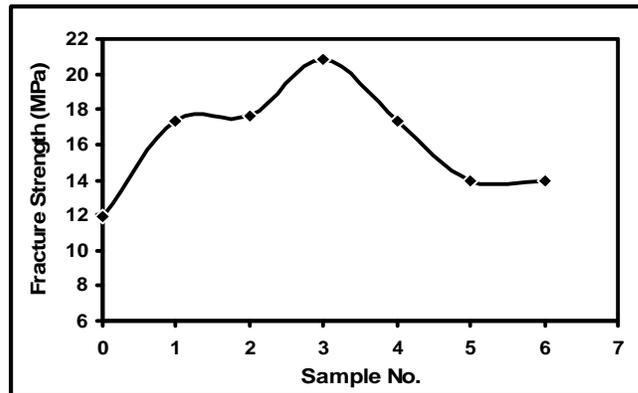


Figure (8) shows the effect of (HA:ZrO₂) addition on fracture strength of sintered dental ceramic batch compact.