Physical-mechanical properties of dental composites by addition oyster shell powder as fillers

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Abstract

Dental composites material prepared in by addition different weight ratio of biomaterial fillers to Calcium-Fluoroaluminosilicate glass. The oyster shell powder was added at weight ratio 3%, 5 %, 7% and 10% into the glass filler as a biomaterial. The physical properties such as the depth of cure, sorption, solubility, flexural strength and Diametric tensile strength were determined according to ISO 4049. The Results showed that adding 3% weight ratio of Oyster shell to Fluoroaluminosilicate glass was led to improved the flexural strength, sorption, and DTS. However, increase a load of oyster shell powder (>5%) indicated a reduction within the mechanical properties of the tested composites.

Keywords: oyster, $CaCO_3$, dental composites, sorption, depth of cure, flexural strength.

1. Introduction

Dental restorative material is considered important material used in teeth restoration which considers an important branch in dental medicine. Composite effected with many circumstances inside the oral environment such as PH of oral environment and eating hot or cold drink and the kind of food [1,2]. However, photic dental composite consisted of three part which is resin polymer and filler which form about 70% of composite content and the coupling agent between the filler and polymer [3]. There are many kinds of filler such as glass filler which considered famous filler used in dental restorative material. Many research tried to develop composite which was focused on (filler) and tried to change filler kind or filler particle size or filler volume or change coupling agent between the filler and polymer [4-10]. There are many types of filler such as chemical filler like glass filler [10], or biomaterial filler like oyster filler which was used in many of research [11,12].

Oyster shells are one of the most common forms in the production "natural source" of Ca supplements, because mainly comprise from about 95% calcium carbonates, followed by material copper, nickel, cobalt, and iron oxide [13,14], furthermore the least expensive [6,15]. The key feature of Calcium Carbonate is porous particles this feature leading to increasing the surface area which allows for increased free surface areas of the inter-pore polymer confinements. This allows improving physical and to some extent chemical surface properties. Furthermore, because of their advantageous and potential properties and good biocompatibility were entered in many medical uses, such as tissue engineering, as scaffolds, bone-tissue, and dental transplants [16,17]. Calcium Carbonate tends to be crystalline and mutual transformations between $CaCO_3$

polymorphs the most common forms calcite, needle-like aragonite, the unstable vaterite [18]. The oysters were availability and cheap price, easily prepared and good biocompatibility with the oral environment. The main objective of this study was to improve and develop the physical and mechanical properties of new kind of bio filler and verification weather fixed to manufacture the composite.

2. Methods and materials

2.1 Preparation the composites

The Calcium-Fluoroaluminosilicate glass (CaFAlSi) and oysters used in this study were conducted synthesized and characterization their properties in the College of Science, University of Basrah [19].

The specific surface area using Brunauer-Emmett-Teller analysis (BET) was founded for CaFAlSi glass and biomaterials which indicated 2.475m^2/g for CaFAlSi glass and 8.659m^2/g for biomaterials, also primary particle size was calculated 0.966μm and 269 nm respectively. The Calcium-Fluoroaluminosilicate (control) fillers and biomaterials (oyster powder) fillers were silanized with a 1.5-3 wt% of γMPS (γ-ethacryloxypropyltrimethoxy), respectively. γMPS was blending for 1 hr in an aqueous solution. The ratio of ethanol to distilled water was 3/1 wt%. (solution acidulated at pH 3.5-4 using few droplets of acetic acid). The treated fillers were dried for over 1 week at room temperature, then the silanated fillers were mixed with organic matrix consisted Bis-GMA and TEGDMA about (30/70) wt% with filler ratio loaded (76%wt.). Table 1 describes the composition of the materials and the ratio of oyster shell used in this study.

Table 1: Compositions of the composites used in this study (wt)%

Materials	(0%)Control	load-displacement of oyster powder			
		Oys3%	Oys5%	Oys7%	Oys10%
Bis-GMA*/ TEGDMA**	24	24	24	24	24
filler : Calcium-Fluoroaluminosilicate	76	73	71	69	67
Oyster shell (%)	0	3	5	7	10
CQ†	0.05	0.05	0.05	0.05	0.05
DMAEM ‡	0.05	0.05	0.05	0.05	0.05

*Bis_GMA: Bisphenol A-glycerolate dimethacrylate, **TEGDMA: Triethylene Glycol Dimethacrylate**,** † Camphorquinone, ‡ Dimethyl aminoethylmethacrylate.

All specimens were inserted to the mold and polymerized using blue LED light unit source (Woodpecker, China) with intensity 600 mW/cm^2 for 40 sec (radiant exposure= 24 J/cm^2) on both sides of the specimens.

2.2 Depth of Cure

Four specimens from each tested composites uncured were condensed into a stainless steel mold (4 mm and 10 mm in diameter and thickness respectively), The ISO 4049 defines the depth of cure, as 50 percent of the length of the composite specimen after the uncured material is removed spatula with a plastic [20].

2.3 Water sorption and solubility

Three specimens were inserted in to stain steel mold with 15mm in diameter and 1 mm thickness to measure water sorption and water solubility. these specimens were polymerized for 40 sec on both sides of the specimens. All new samples were placed into desiccator at 37°C for 24hr's and weighted (m_0) this proses repeated, so when samples placed in distilled water at 37°C we named that weight (m_1). After an interval of about 180 days, specimens removed from the water and entered at silica gel solution for a week (m_2). Water sorption (W_A) and water solubility (W_S) were calculated from [21]:

$$w_A = \frac{m_1 - m_2}{V} \quad (1)$$

$$W_S = \frac{m_0 - m_2}{V} \quad (2)$$

2.4 Flexural Strength and Flexural Modulus

In this measurement, used stain steel mold (25mm,2mm,2mm) in length and thickness and height to measure mechanical properties (flexural strength and flexural modulus). Three specimens of each ratio were made. All specimens were tested with overlap method and using the three-point bending test was performed on the specimens using a universal testing machine (Zwick/Roell BT1FR2.5TN Germany) at a crosshead speed of 1 mm/min. all specimens placed in distilled water for 24hr's. Flexural strength and flexural modulus were calculated using:

$$F_S(MPa) = \frac{3pL}{2bd^2} \quad (3)$$

Where p the failure load (N), L the distance between the supports (20 mm), and b and d, the width and thickness respectively of the specimens (mm).

$$F_M\ (MPa) = \frac{3FL^3}{4bd^3D} \quad (4)$$

Where F: force at deflection, L distance between two points (20mm), b width, D cutting limit [22].

2.5 Diametric Tensile Strength (DTS)

For diametric tensile strength measurement, three specimens were prepared and placed in to split stainless steel mold (3mm, 6mm) in diameter and height respectively and polymerized under 480nm blue LED for 40 sec on both sides of the specimens. then all new samples were removed from the water and tested using. A universal testing machine (Zwick/Roell BT1FR2.5TN, Germany) with a crosshead speed of 1 mm/min. The DTS (MPa) was calculated as [23] :

$$DTS\ (MPa) = \frac{2p}{\pi DL} \quad (5)$$

Where p load at fracture, D diameter of specimens, L specimen's length respectively.

3. Result and Discussion

Figure 1 shows the DOC (mm) as a function of the weight ratio of the oyster. From the figure, noticed that the varying in DOC was not large when adding oyster. Although, the oyster is useless material for the environment and we can use as a dental material to restore the teeth.

The mean values (Std. Deviation) results of sorption, solubility , depth of cure, flexural strength and diametric tensile strength for tested composites presented in Table 2, the depth of cure (DOC) of tested composites was measured according to ISO 4049:2009 which ranged at 2.91±0.18 to 2.32±0.30 mm, also the result denoted decrease with DOC after added oyster filler when compared with control composites, Oys3% a slightly low with the value of control (CaFAlSi filler) and lowest Depth of cure returned to Oys5%. the reason that behind this decreased value of the depth because oyster shell ($CaCO_3$ particle) filler considered turbid and non-transparent powder, which can form a barrier to prevents the passage of light, and the other reason It is possible to distribution particles and not distribute them regularly within specimens of composites which cause scattered the light and not reaching a deeper depth.

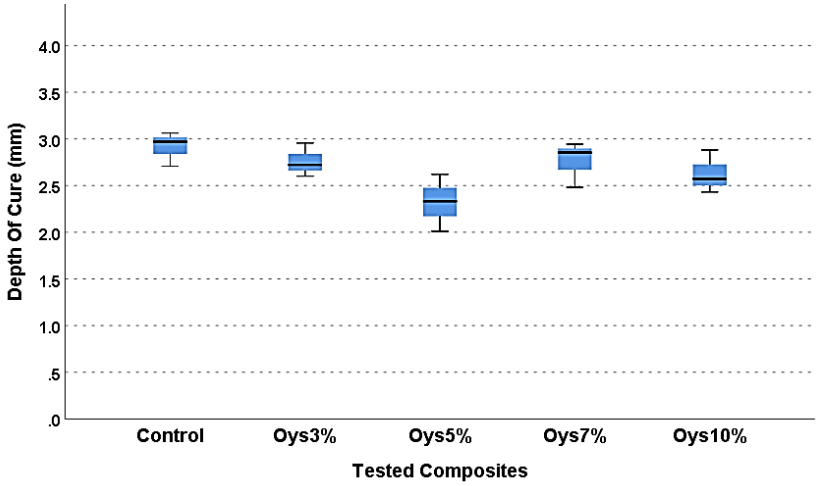

Figure 1. The depth of cure (mm) as a function of weight ratio of oyster in the glass.

Table 2. Mean values (Std. Deviation) properties of tested composites.

Tested Composite	DOC (mm)	sorption ($\mu g/mm^3$)	solubility ($\mu g/mm^3$)	FS (MPa)	FM (GPa)	DTS (MPa)
CaFAlSi	2.91 (0.18)	28.92 (5.48)	6.12 (2.05)	64.90 (10.77)	13.00 (1.4)	50.73 (6.07)
Oys3%	2.76 (0.18)	25.58 (6.60)	5.04 (0.9)	79.83 (17.87)	13.57 (1.19)	51.13 (5.65)
Oys5%	2.32 (0.30)	30.59 (3.44)	5.20 (0.58)	48.35 (16.08)	13.42 (1.09)	36.81 (4.85)
Oys7%	2.76 (0.24)	26.33 (4.09)	3.77 (1.2)	46.51 (7.27)	13.36 (0.37)	31.85 (9.42)
Oys10%	2.65 (0.11)	29.25 (4.43)	3.66 (0.71)	44.85 (2.84)	13.12 (0.56)	32.02 (2.76)

Figure 2 shows the water sorption (μg/mm^3) as a function of weight ratio of oyster in the glass. Water sorption was lower than the control values recorded except the value of composite load 5% (Oys5%).

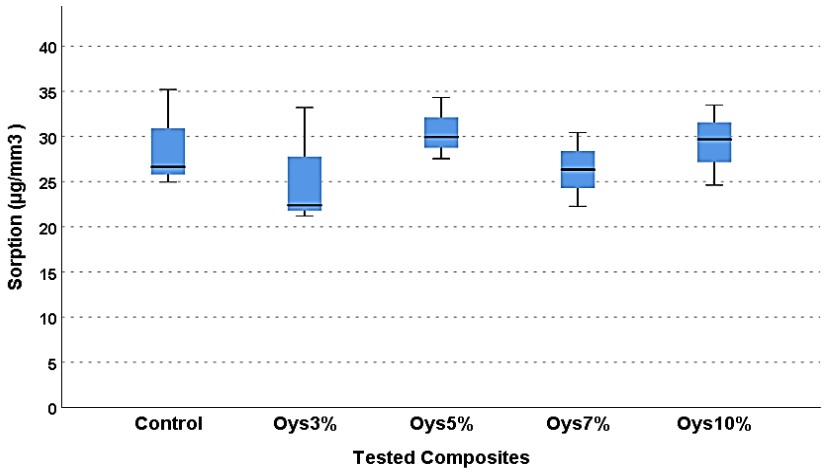

Figure 2. Water sorption as a function of weight ratio of oyster in the glass.

Water sorption and water solubility were measured according to the ISO 4049:2000 which specifies that sorption must be <40μg/mm^3 and solubility <7.5 μg/mm^3. the results of water sorption for tested composites was ranged 30.59±3.44-25.58±6.60 μg/mm^3, which is less than control (CaFAlSi) sorption value (28.92±5.48μg/mm^3) except for Oys5% recorded the highest water sorption rate. The slight decrease in absorbance is due oyster (Calcium carbonate) considered a porous material so it had the ability to absorb a high amount of water, the second reason diffusion of ionic salt inside crystal structure of $CaCO_3$. Some of the research said that the main cause of sorption of the material related to small particle molecular of the water which is about 0.158 nm. Sorption of the composite must be little because if they are large, the water molecular will try to break down the bond (silane coupling agent) between filler and polymer which reduce the age of the composite.

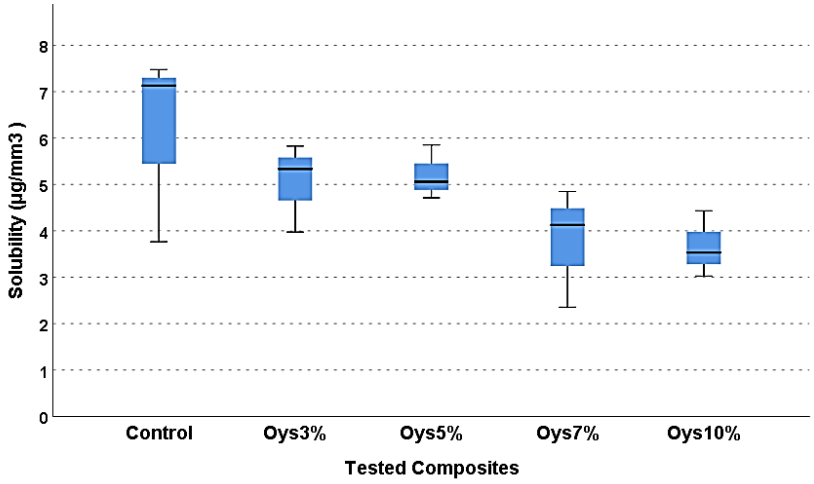

Figure 3. Water solubility as a function of weight ratio of oyster in the glass.

Water solubility value of the tested composite was shown in Figure 3. From the figure shows the range between 5.20±0.58 and 3.59±1.62µg/mm^3. These values are lower than the value of control composites 6.12±2.05µg/mm^3 and a decrease of solubility proportional with the increasing of oyster shell powder ratio in the filler rates (except for Oys5%), the reason for this small value because calcium carbonate is considered very low solubility soluble in water.

The tested composite flexural strength value for all ratio shown in Figure 4. There is a significant increase between the flexural strength of Oys3% and control composite.

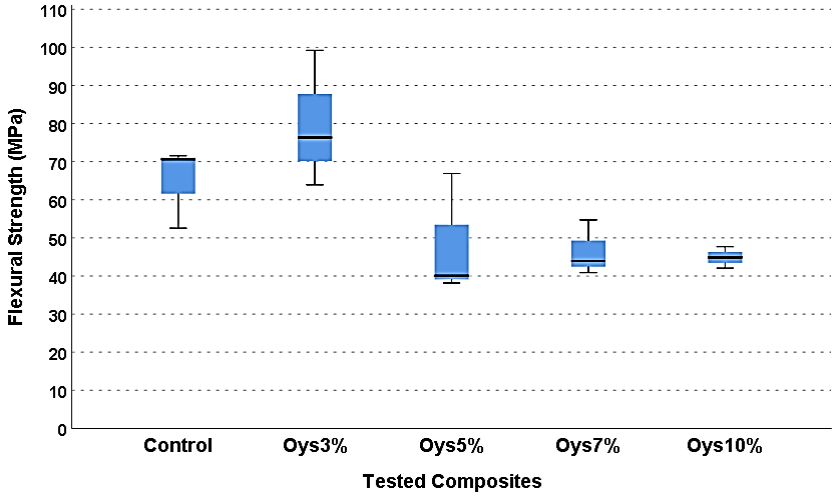

Figure 4. Flexural strength (MPa) as a function of weight ratio of oyster in the glass.

The flexural strength of oyster powder filler ratio was calculated and ranged 79.83±17.87 to 45.66±3.25 MPa. Oys3% denoted the highest value of the flexural strength and also more than the value of control composite, however, this increase quickly fell and collapsed at the high ratio of oyster powder filler. Where it recorded Oys10% lower value. This is because of $CaCO_3$ (oyster shell) consider as porous material which gives a high surface area which causes and give a good bond between filler and matrix, this bond is good and gives good results in improving the value of flexural strength in the low ratio of oyster powder ~ <3%, but is soon negative effect when the ratios increase to >5%. The result of value was an acceptable value for flexural strength according to the ISO 4049:2000 are (50-80) MPa, so our value considers a good and high value of oyster. Also, the flexural modulus depends on the transition of the stress between the filler and the matrix in the composite [24]. This transition of stress depends on a good bond on the matrix with filler and penetration into its porous, Figure 5 shows the flexural modulus as a function of oyster weight ratio in the glass, it is indicated to the significant difference in elasticity coefficient values in vehicles after load biomaterials filler to composites. The flexural modulus of load-displacement oyster composites was range from 13.57±1.19 to 13.12±0.56 GPa, there are no obvious differences in values flexural modulus in tested composites in this study, which reveals that the modulus has not been affected by the oyster shell when adding to fillers.

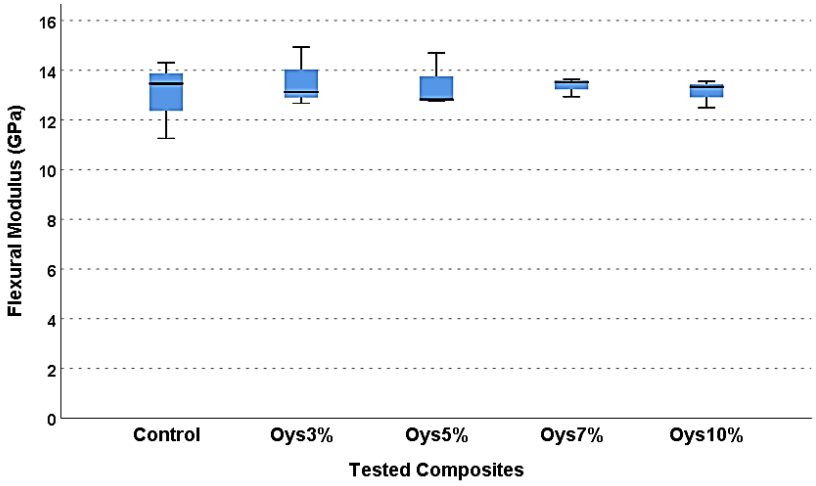

Figure 5. Flexural modulus (GPa) as a function of weight ratio of oyster in the glass.

Figure 6 show the diametric tensile strength (DTS) as a weight ratio of oyster in the glass, the breakdown of the tested composites shows after a 3%wt. The DTS test was described behave the material as a brittle material under compression forces [25], furthermore, DTS is one of most and common in dental materials and more important than compressive strength because this material will more likely be subjected to tensile or shears than in compression in clinical application [26]. The value of DTS in this study ranged from 51.13±5.65 to 31.85±9.42 MPa, tested composites with increase load-displacement behavior as the brittle pattern and decreases the value of DTS. DTS value for dental composites in range (30-55 MPa) which shows that our value for tested composites was acceptable.

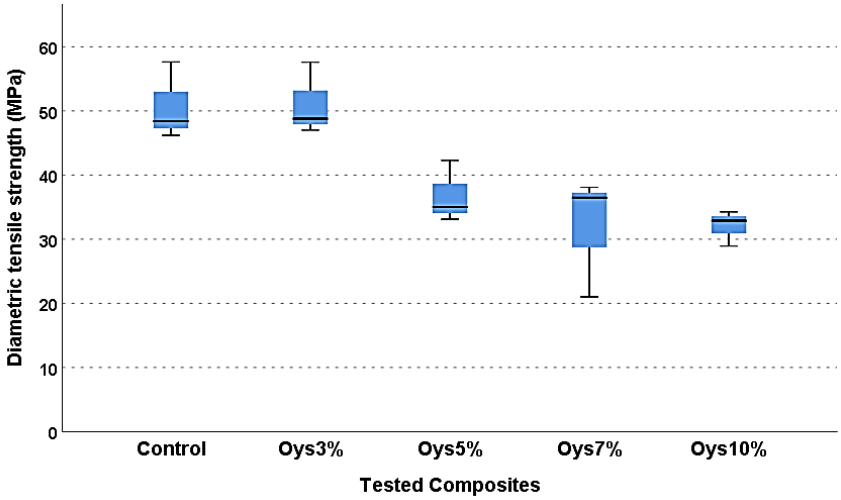

Figure 6. DTS (MPa) of tested composites loading 0-10 wt.% of oyster shell.

4. Conclusions

The results can evaluate the effect of adding an oyster to filler dental composites on physical and mechanical properties. There is improvement in the properties for ratio

(3 wt%) of oyster . The sorption and solubility are less than control. The flexural strength increased significantly, but did not affect the elasticity coefficient and the depth of cure. we are not recommended to add a high ratio (up to 5%) of oyster powder to filler, because the results were not encouraging. Therefore, load-displacement of oyster powder as fillers can be considered as an important and applicable way to reinforce dental composites.

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الخواص الفيزيائية والميكانيكية لمتراكبات الأسنان باضافة مسحوق مالئات القواقع

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المستخلص

تم تحضير متراكبة ترميم الاسنان عن طريق اضافة نسب وزنية مختلفة (3%, 5% ,7%)ونسبة (10%) من مالئات القواقع الى متراكبة الزجاج (كالسيوم فلوروالومينوسيلكات) .وتم حساب الخواص الفيزيائية (عمق التبلمر , الامتصاصية , الذوبانية) والخواص الميكانيكية (المطاوعة , معامل المرونة , الانضغاطية) وحسب نظام الجودة العالمي الخاص بمواد ترميم الاسنان . وبينت النتائج ان اضافة نسبة 3% من مالئات القواقع الى متراكبة الزجاج ساهم وبشكل كبير في تحسين خواص المتراكبة كالمطاوعة والانضغاطية والامتصاصية .كما واظهرت النتائج ان زيادة محتويات المالئات ادى الى تقليل جودة الخصائص الميكانيكية التي تم اختبارها على المتراكبات