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Research Article

Mechanical Properties and Linear Shrinkage of Resins Reinforced with Micro Hydroxyapatite for Dental Restoration

Mohammed H. Al-Maamori[Á*], Samir Hamid Awad[Á] and Anas Kareem Hussein[Á]

[Á]College of Materials Engineering, Babylon University, Iraq

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Abstract

In this study, hydroxyaptite (HA) $Ca_{10}(PO_4)_6(OH)_2$ nano size has been synthesized from waste materials (egg shell and fish bone) and then mixed with resin composite in order to measure the mechanical properties and linear shrinkage of composite materials designed for dental restoration. The material was composed of visible light-curing monomer (composan LCM) as a matrix and hydroxyapatite (nano particles) as a reinforcing filler at different ratios (5-10-15wt%).The results show that the mechanical properties increases with added HA powder and up to 287Hv at the rate 15% of hydroxyapatite as well as the case for the compressive strength and the depth of cure to 130 Mpa and 2.65mm respectively, while the linear shrinkage decrease to 0.097mm at 15% hydroxyapatite.

Keywords: *Micro Hydroxyapatite, Mechanical properties etc.*

Introduction

Composite resin is a common material used for clinical oral filling and bonding, in which a large number of inorganic fillers play an important role in the mechanical and biological properties of composite resin (Guo et al,2010). Polymer-matrix composites are the most widely used dental restorative materials, whether after caries, fracture, or endodontic therapy, given their physico-mechanical properties. Among a wide range of such materials, those based on Bis-phenol A-glycidyl methacrylate (Bis-GMA) and related moieties are the most commonly used, having relatively low thermal expansion and shrinkage, low volatility, and clinically acceptable handling properties . However, their durability and strength are considered insufficient, and they lack bioactivity and biocompatibility (Hongquan ,Brian ,2012). Also, polymerization shrinkage of dental resin composites is one of the main problems for clinicians during the restoration. Hence, any data on the shrinkage of such dental composites could contribute for a successful and proper restoration (Bilge et al ,2010). Such Polymerization shrinkage of resin composites remains a clinical concern due to the associated residual stresses that are thought to play a role in marginal failures, micro leakage and recurrent caries . Shrinkage stress may also induce tooth deformation and cohesive failures within the restorative material or dental structure, which can lead to postoperative sensitivity (Alessandro et al ,2004).

Therefore; a dental resin reinforced with dispersed HA crystals seems, in principle, a favorable restorative materials for human tooth, as it is for bone tissues. The use of HA in restorative dentistry offers several promising advantages ,including intrinsic radio-opaque response, enhanced polish ability and improved wear performance ,since synthetic HA has a hardness similar to that of natural teeth (Raul et al ,2002).

Experimental procedure

1. Preparation of Hydroxyapatite micro particles

1.1 Preparation from egg shells

To prepare micro HA powder from egg shells the following steps were followed:

- Egg shells were collected and their surfaces were mechanically cleaned.
- The raw egg shells were calcinated in an air atmosphere at 900°C using the furnace.
- The thermal treatment through the calcinations had two parts: in the first 30 minute, most of the organic materials were burnt out, whereas in the second part the egg shells transformation into calcium-oxide was obtained (holding time was 3 hour).
- To synthesize calcium phosphate powders, shells were crushed and milled in a ball milling, which equipped with alumina balls and bowls.
- The crushed egg shell was reacted by an exothermic reaction with phosphoric acid (wt% shell: H3PO4-50:50).
- The mixtures were milled for 10 hour at 350 rpm (a planetary ball milling), for homogenous mixing and to prevent agglomeration of the calcined.
- After milling, HA powders were heat treated at 900 °C for 2 hour in air atmosphere using the calcinations furnace.

*Corresponding author: **Mohammed H. Al-Maamori**

1.2 Preparation from Fish bones

To prepare HA powder from fish bones the following step were carried out:

- The fish bones were boiled in distilled water for 1hour and washed using a strong water jet to eliminate the fish meat.
- The washed fish bones were then dried and heated at 500°C for 30 minute using the furnace
- It is then crushed in crusher.
- The powder size obtained after crushing was in micron. This was further reduced through planetary ball milling process.

After that, XRD test was conducted to identify the powder, and the particle size analyzer were involved to characterize and calculated particle size.

2. Resin materials and treatment by Halogen Light

Resin selection was done using light curing compound material type (composan) LCM/ (PROMEDICA Domagkstr 24537 Neumunster \(Germany), corresponds to EN 24049/ISO 4049. Composan LCM is polymerized by halogen light (blue light) with (250 mw) intensity source of halogen light. The all samples were treated for 15 sec.

Results and Discussion

1-1XRD result of egg shell-HA powder

The shell of egg has been scanned with XRD diffractometer in diffraction angle from 10º to 50º and this stated in fig. (1). It also can be observed that the highest peak intensities were belonged to the HA peaks ,thereby, proved the success of egg shells process in producing the required HA powder.

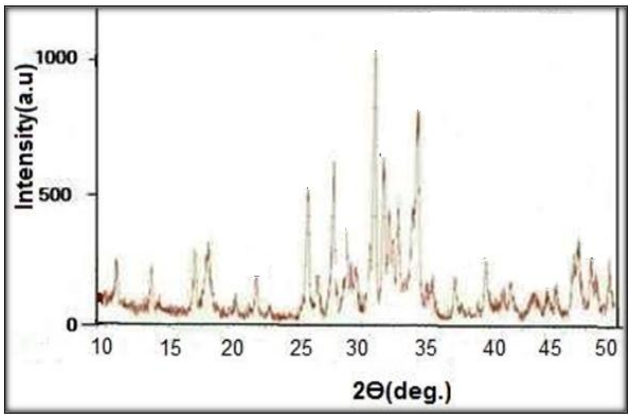

Fig.1 XRD patterns of egg shell - HA powder.

1-2XRD result of fish bones-HA powder

The XRD patterns of fish bones powder diffracted at angle 10º to 50º are shown in fig.(2). The patterns showed that all peaks were pure HA after comparison with standard XRD card NO.(09-0432). This results proved the success of fish bones method in producing pure HA powder.

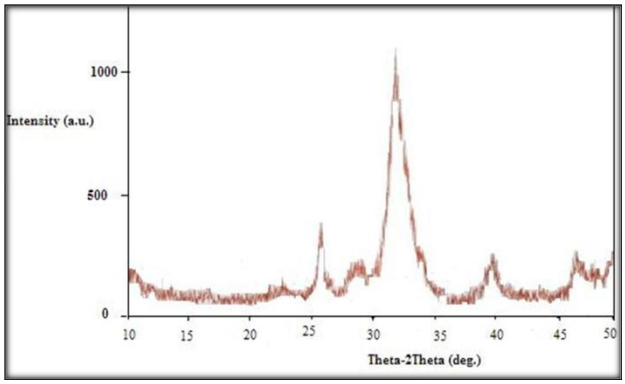

Fig. 2 XRD patterns of fish bones-HA powder.

1-3Particle size analyzer

Fig.(3), (4), show the particle distribution of micro –HA. It can be seen that more than 85% of the particles are between 1 µm and 19.5 µm with a mean size of 6.5 µm as shown in fig.(3), and the particles distribution shown in fig.(4) are between 0.3 µm and 20µm with a mean size of 4µm,

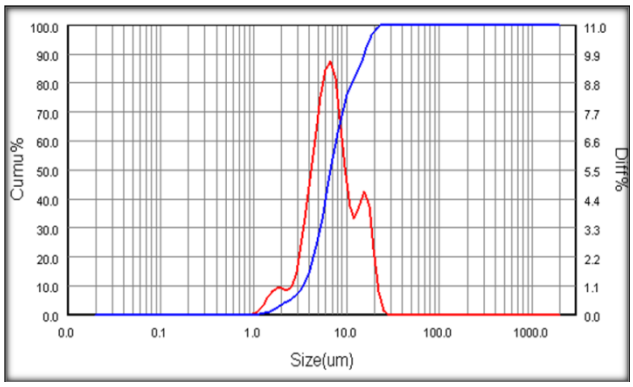

Fig. 3 Particle size analysis of egg shell HA-powder

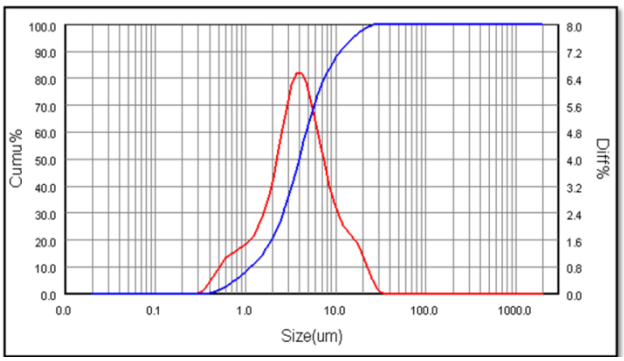

Fig. 4 Particle size analysis of fish bones HA-powder

1-4 Hardness and compressive strength test

Fig.(5) &(6) showed the results from hardness compressive strength test. It is obvious that the hardness and compressive strength increased with increasing of HA additions due to high mechanical properties of the HA powder.

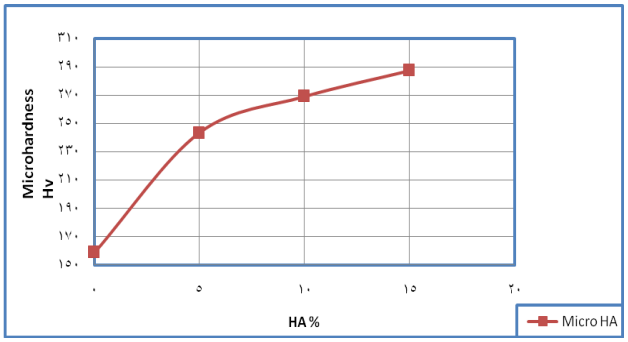

Fig. 5 Effect of micro HA addition on the hardness, When (wt) means weight percent

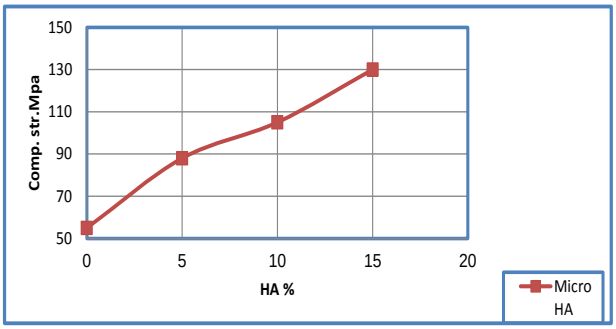

Fig. 6 Effect of micro HA addition on compressive strength.

1-5 Depth of cure and Linear shrinkage

Fig.(7) shows the results from testing of the depth of curing. It can be observed that the depth of cure (DOC) increased slightly with increasing the addition of micro HA due to the addition of HA powders at the ration used in our work. The micro HA addition could record high DOC. The reasons behind such behavior attributed to presence of micro HA particles which facilitated light penetration and ,thereby, increasing DOC ,where , light scattering is related to filler particle size . Furthermore, small sizes of nano particles provided more difficulties in light penetration.

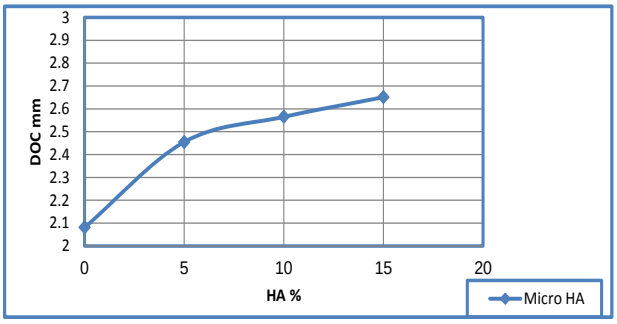

Fig. 7 Effect HA powder on the Depth of cure.

Fig.(8) shows the results from linear shrinkage measurement . It can observed that the linear shrinkage decreased with micro HA increased in the matrix due to the fact that the higher inorganic content is associated with lower polymerization values which are in direct relation with reduction the shrinkage. Furthermore , the filler particles act as an impediments to the polymerization reaction.

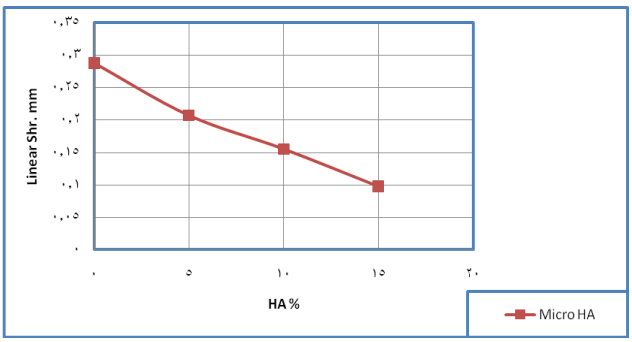

Fig. 8 Linear shrinkage of micro HA-resin composite

1-6 Scanning electron microscope (SEM)

SEM was used to study the dispersion and distribution of the micro fillers in the resin matrix. Fig. (9) and (10) show SEM images of pure matrix ,15% micro HA-resin. In the fig. (10), the micro structure shown well dispersion, homogenous distribution of micro HA in the resin matrix, relatively, without agglomerates.

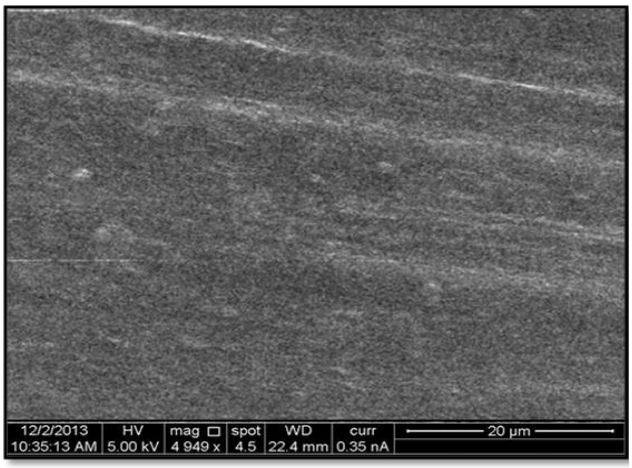

Fig. 9 SEM image of the pure matrix

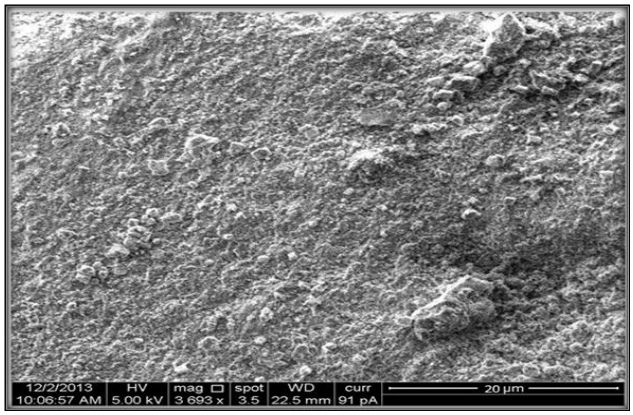

Fig. 10 SEM image of 15% micro HA-resin composite

Conclusions

Results showed that the mechanical properties such as hardness, compressive strength and depth of cure improved with the addition of HA% but linear shrinkage decreases with added and this property is good for improving dental fillings.

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