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Manufacture and Characterization Physical and Mechanical Properties of Bioveneer Based Ark Clam Shells Composite

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ABSTRACT

Composite materials have been created for the applications of dental veneer from a mixture of raw materials : hydroxyapatite (HAp) powder from ark clam shells and epoxy resins by cold compression method with variations of composition hydroxyapatite (HAp) powder : epoxy resin (75 : 25)%wt, (80 : 20)%wt, (85 : 15)%wt, (90 : 10)%wt, dan (95 : 5)%wt. Sampling is created in three steps. The first step of ark clam shells powder is treated by smoothing it with a particle size of 200 mesh and heated at 900°C for 2 hours after it had been synthesized into hydroxyapatite (HAp) powder. The second step of the filler (hydroxyapatite powder) is mixed with dry mixing and then mixed with epoxy resin as a matrix. The third step of the homogeneous mixture is then inserted into the mold and compacted by heat to be more dense with pressure of 300 MPa or 3 ton held for 5 minutes at 27°C. The characterization results showed that the optimum composition of hydroxyapatite powder: epoxy resin (75 : 25)%wt with density value 0,1185 x 10^3 kg/m^3, water absorption 1,163%, composed of -PO_4 (phosphate), -CO_3 (carbonate) and HOH groups. Mechanical properties with fracture strength 116 MPa, tensile strength 66,12 MPa, modulus of elasticity 3297,99 MPa and brinell hardness 75,87 HBR whose characterization results have met the conventional dental veneer standards.

Keywords: Clam Shells Powder, Dental Veneer, Epoxy Resin, Hydroxyapatite

I. INTRODUCTION

The development of science in the field of material, especially physics found a way to restore abnormality or tooth decay, especially related to aesthetics and also to assist in the field of dentistry, one of which is by coating the surface of the tooth with the material is veneer[1]. The veneer is a thin, slightly transparent tooth layer applied to the surface of the facial and proximal parts of the tooth permanently using acid etching and the bonding agent. Veneer restorations can be either direct or indirect restorations, and can be made with composite or ceramic resins. Direct veneer restoration using composite resin is often done by dentists, but it requires high skills in forming good morphology. The disadvantages of this restoration are easy to wear and fracture and shrinkage during polymerization which can cause edge leakage. Whereas resin composite indirect veneer restoration requires strong attachment to the tooth surface so that the veneer restoration is not separated easily[2][3].

Behind all the benefits obtained from the use of veneers, there is some controversy in its use because nowadays there is widespread use of veneers which are only for aesthetic purposes without dental problems. This tends to be detrimental to healthy teeth, because before veneer is applied it is carried out in advance on tooth enamel[4] [5]..

This research was made to find out the best way to make veneers that produced have good aesthetics but still be healthy, namely making veener from organic matter in the form of ark clam shells reinforced epoxy resin modified by biocomposite. Where the ark clam shell are used as powder with the ball mill method to reduce the size of the crystal so that the density will be higher and strengthen the veneer matrix bond. The feasibility of shells as biocomposite fillers is due to local wisdom waste, which has a compound $CaCO_3$ of 67.55% and the remainder is the content of SiO_2 and Al_2O_3 so that it has the characteristics to form strong covalent bonds[6][7] [8]. While epoxy resin is a polymeric material which is used as a matrix that have a characteristic of density 1150 kg/m^3, a tensile strength of 65-79 MPa and an modulus of elastiscity 3100 MPa so that it can increase the strength of biocomposite as a bioveneer application.

II. METHODS AND MATERIAL

A. Equipment and Materials Research

The equipments used are spatula, beaker glass, oven, caliper, digital balance, moulding sample from iron, universal testing machine, sieves of 200 mesh, mortal, erlenmenyer tubes, burettes, indicators of universal, ovens, funnels and filter paper, hot press hydraulics, hardness brinell machine, scanning electron microscopy (SEM) EDX, fourier transform infrared (FTIR) and stopwatch. The materials used are ark clam shell powder, epoxy resin, alginate, aquadest, HCl 37% and NH_4OH

B. Research Variables

Research variables on the manufacture of bioveneer include raw material composition and characterization.

TABLE I
PERCENTAGE OF BIOVENEER BASED ON HYDROXYAPATITE REINFORCED EPOXY RESIN

Sample Code	Hydroxyapatite Powder (%wt)	Epoxy Resin (%wt)
A	95	5
B	90	10
C	85	15
D	80	20
E	75	25

As for the characterization of hydoxyapatite from ark clam shells powder materials such as functional group. While the characterization of bioveneer include : physical properties (density and water absorption), mechanical properties (hardness strength, tensile strength, modulus of elasticity and modulus of rapture) and surface morphology and elemental content.

C. Research Procedures

- **Preparation of $Ca(OH)_2$ powder from ark clam shells**

The ark clam shell waste was obtained from the Helvetia Market, is soaked first to remove dirt and odor on the ark clam shell then cleaned and finally washed with distilled water. After drying, it is smoothed using a filtered collision using a sieve of 200 mesh. Then the shells that have been filtered are then in the furnace up to 900°C for 2 hours so that CaO powder is formed. When the CaO powder weighed of 10 grams of ark clam shell. Put in a glass beaker then add 1 M HCl as much as 200 ml stirred with 700 rpm until dissolved. And NH_4OH is added to a base solution of pH 12 to form a $Ca(OH)_2$ solution.

- **Preparation of hydroxyapatite from $Ca(OH)_2$ ark clam shell powder**

Provided a $Ca(OH)_2$ solution that has been produced. Titrated with 0.3 M H_3PO_4 solution at a speed of 1 ml/sec by using a burette. Stirred at a speed of 700 rpm for 3 hours. Regulated with pH by adding NH_4OH so that the base solution is pH 10. Let stand for 15 hours until the HAp deposit is formed. Filtered the resulting sludge. Washed sediment with hot distilled water to remove Cl^- (chlorine) ions. After it, the sediment dried in the form of hydroxyapatite (HAp) in the oven, then filtered at a temperature of 1000°C. The results of deposition of HAp were characterized using FTIR to determine the hydroxyapatite functional groups produced.

- **Manufacture of bioveneer based on hydroxyapatite/ epoxy resin composite**

The basic ingredients which have been in the form of powder namely, ark clam shell powder as filler and epoxy resin as matrix is weighed in advance with variations in the composition seen in Table 1. The ark clam shell powder mixing with epoxy resin by using a wet mixing method with a total mass of 30 grams which is then stirred it for 1 hour by using a spatula in a beaker glass. After the materials are evenly mixed, then pour the ingredients that have been mixed into the mold that has been smeared with vaseline so that the dried sample is easy to remove. Compressed at a pressure of 300 MPa with a holding time of 5 minutes. Issued a compacted sample from the mold formed square with a length of 3 cm, width 3 cm and thickness of 3-4 mm then dried and characterized veneer samples.

III. RESULTS AND DISCUSSION

A. Characterization of Functional Groups of HAp (Hydroxyapatite) in Ark Clam Shells

- **FTIR (Fourier Transform Infrared)**

FTIR analysis was performed by using the Nicolet IS10 to determine the functional groups in the synthesized compound and determine the number of components in the sample indicated by the presence of a peak at a particular wave number[7]. This characterization is carried out to obtain information about the vibrations of phosphate, carbonates and amide compounds, and to ensure the synthesis of ark clam shell hydroxyapatite compounds without the association of organic groups.

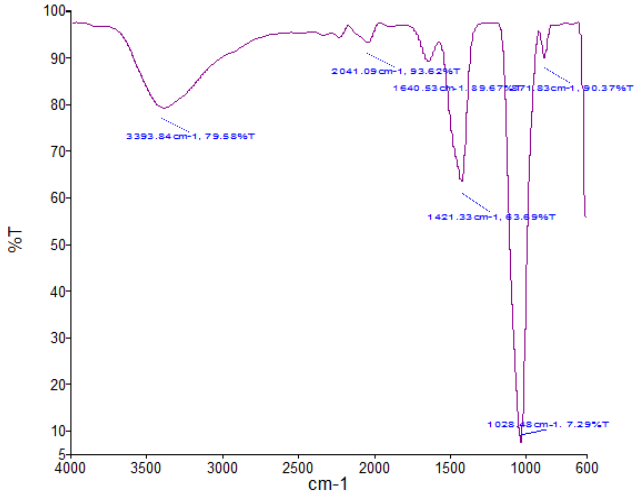


Figure 1 : The functional group analysis of hydroxyapatite (HAp) from ark clam shell powder

The results of FTIR analysis showed that the hydroxyapatite (HAp) samples of blood clam shells have PO_4^{3-} groups which is the highest intensity detected at wave numbers 1028.48 cm^{-1}. This value indicates the presence of P-O vibration from the PO_4 group. The OH^- group is indicated by the moderate intensity of the wave absorption band detected at wave number 1640.53 cm^{-1}; 2041.09 cm^{-1}; 3393.84 cm^{-1}; this value indicates the presence of hydrogen bonds with the vibration of the H-O-H function group. While the absorption band at wave number 1421.33 cm^{-1} shows CO_3^{2-} groups which indicate the presence of C-O vibrations from the CO_3 group. The functional groups contained in each sample according to the characteristics of hydroxyapatite from the FTIR spectrum are PO_4^{3-}, OH^-, CO_3^{2-}, and HPO_4^{2-}, which are non-stoichiometric characteristics of hydroxyapatite[8][9].

B. **Physical Properties of Hydroxyapatite (HAp)/Epoxy Resin Composite as Dental Veneers**

- **Density**

Density test is carried out to measure the density of atoms making up a material that binds to one another or interacts between one atom and another by measuring the mass of each unit of material volume[10].

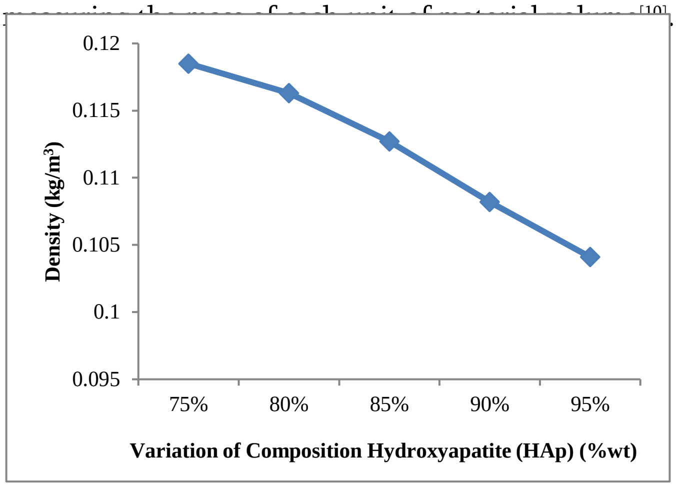


Figure 2 : Test result density of bioveneer based HAp/epoxy resin on various variations composition

The results showed that the best condition of dental veneers was obtained in the composition of hydroxyapatite (HAp) of ark clam shells: epoxy resin (75: 25)%wt. This composition is able to produce a density of 0.1185 x 10^3 kg/m^3. The worst variation conditions occur in the composition of hydroxyapatite (HAp) : epoxy resin (95: 5)%wt with a density of 0.1041 x 10^3 kg/m^3. The results of the dental veener density test in this study have met the conventional dental veneer standard from the results of Stratasys test, namely with an interval of density values of (1.17 - 1.18) x 10^3 kg/m^3. The density value is influenced by the addition of hydroxyapatite (HAp) so that when the air cavity arises due to stirring and compacting processes that cause adhesion-cohesion interface of hydroxyapatite (HAp) with an imperfect epoxy resin matrix which has an impact composite bond becomes weak. So that with the addition of hydroxyapatite (HAp) which has an arrangement of elements of calcium and phosphate which has a free group in the form of H-O-H, P-O, and C-O so that the group will reach and bind the empty cavities to form strong and tight covalent bonds[10][11].

- **Water Absorption**

Water absorption is the ability of a material to absorb water, this absorption power is very important in the manufacture of dental veneer material which knows the optimum absorption capacity of the composite.

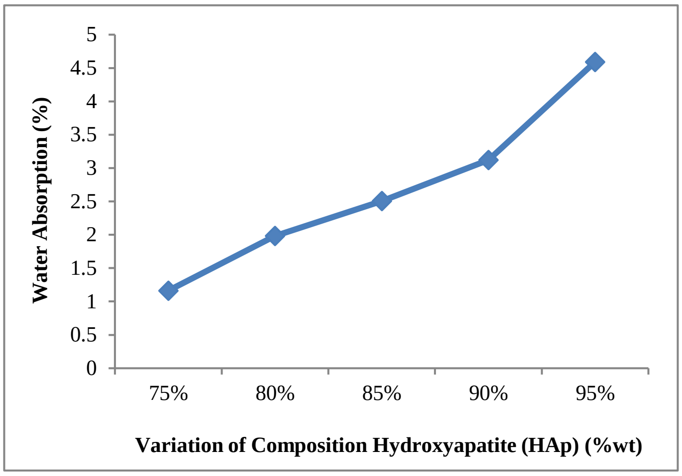


Figure 3 : Test result water absorption of bioveneer based HAp/epoxy resin on various variations composition

From Figure 3, it can be seen that the optimum absorption of dental veneer water occurs in the composition of hydroxyapatite (HAp) : epoxy resin (75: 25)%wt with water absorption 1.163%. While the low absorption of water in the composition of hydroxyapatite (HAp) : epoxy resin (95: 5)%wt with a value of 4,591% water absorption because the composition of the filler is so large that the composite bond by the matrix (epoxy resin) cannot reach all surfaces composite so that the bond becomes imperfect which results in the compaction process, the particles arrange the dental veneer are very weak due to the oxygen molecules entering and forming cavities. The test results for the absorption of dental veneer water in this study have met the standards of conventional dental veneer materials with water absorption of 1.2% - 1.5% at water damping temperatures of 25°C. Water absorption is influenced by the nature of hydroxyapatite (HAp) having a phosphate active group (- PO_4) and carbonate group (- CO_3) which has a brittle nature which is reinforced by epoxy resin which has a hydroxyl group (HOH) which becomes water resistant (watertight) [12]

C. **Mechanical Properties of Hydroxyapatite (HAp)/Epoxy Resin Composite as Dental Veneers**

- **Modulus Of Rapture**

Modulus of rapture test is a test conducted to know the ability of dental veneers to hold the load or horizontal mechanical force that is given until the raapture occurs.

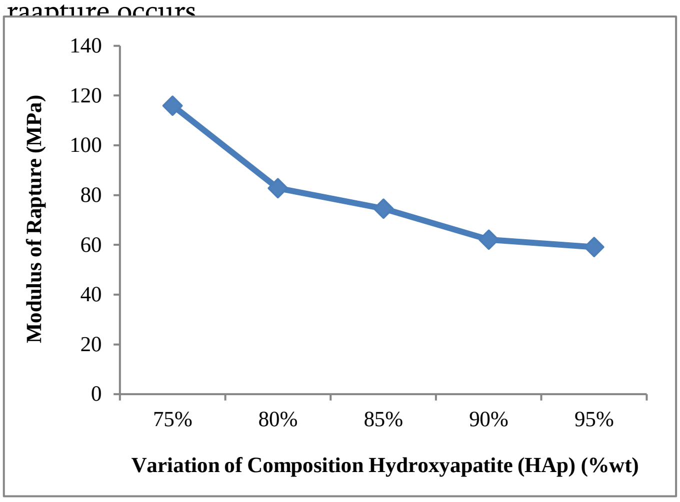


Figure 4 : Test result modulus rapture of bioveneer based HAp/epoxy resin on various variations composition

The results showed that the optimum condition obtained the optimum fracture strength in the composition of hydroxyapatite (HAp) : epoxy resin (75: 25)%wt which is 116 MPa and low conditions in the composition of hydroxyapatite (HAp) : epoxy resin (95: 5)%wt with a fracture strength 59.12 MPa and according to the standard, with fracture strength in conventional veneers of (75 - 110) MPa.

The increase in rapture strength is not only influenced by the micro structure of the material, which includes cavities and cracks formed during the cold compaction process that forms air bubbles (oxygen trapped), but also influenced by the nature of the constituent material. The density of dental veneer samples can affect this because of the homogeneity of the mixture, so that the distribution of hydroxyapatite (HAp) is not evenly spread to the surface of the epoxy resin which results in cracking of the veneer when given an external force[13].

- **Tensile Strength**

Tensile strength test is the ability of a material to hold the load or mechanical force that is given until it is broken.

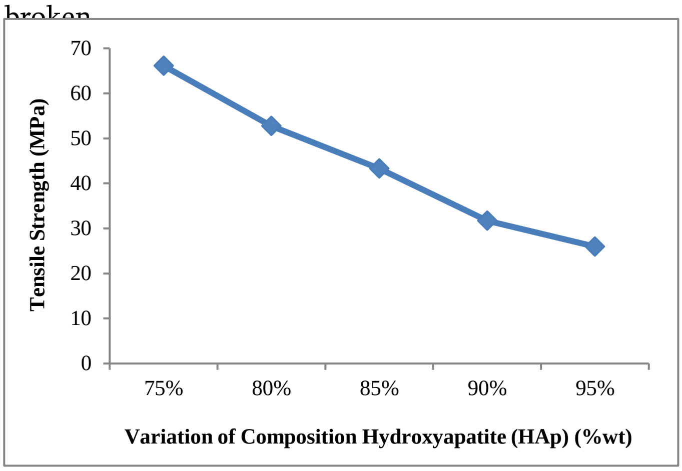


Figure 5 : Test result tensile strength of bioveneer based HAp/epoxy resin on various variations composition

From the results of the graph above, it shows that the optimum conditions obtained good tensile strength in the composition of hydroxyapatite (HAp) : epoxy resin (75: 25)%wt which is 66.12 MPa and less optimum conditions in the composition of hydroxyapatite (HAp) : resin epoxy (95: 5) wt with a tensile strength value of 25.97 MPa with conventional dental veneer standards having a tensile strength of 54-65 MPa. The value of tensile strength decreases when it increasing the filler mass of hydroxyapatite (HAp). This is because the hydroxyapatite (HAp) contains calcium phosphate which has a free group such as phosphate groups (-PO_4) and carbonate (-CO_3) which form covalent bonds to achieve a perfect bond between epoxy resin surfaces with composers of dental veneer composites[14][15]

- **Modulus of Elasticity**Modulus of elasticity is a test that aims to determine how resistant a veneer material is to strain to be more elastic deformation when given vertically outside stress.

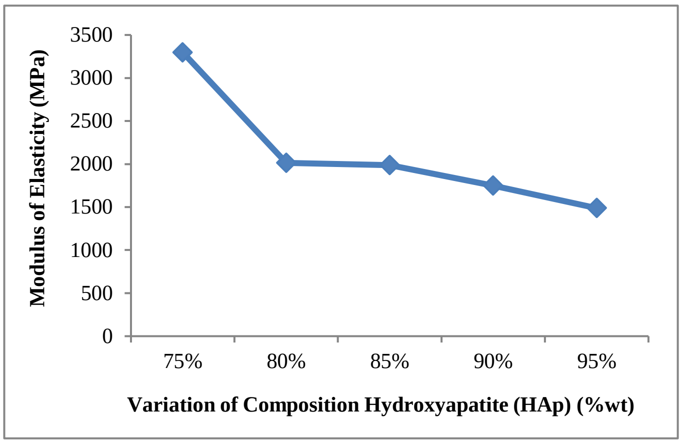


Figure 6 : Test result modulus elasticity of bioveneer based HAp/epoxy resin on various variations composition

Figure 6 shows that the optimum condition obtained a good elastic modulus in the composition of hydroxyapatite (HAp) : epoxy resin (75: 15)%wt which is 3297.99 MPa that has met the conventional dental veneer standard, namely modulus of elasticity 2000 - 3300 MPa. Modulus of elasticity is influenced by the process of making with conventional technique and cold press which is manually stirred with a spatula allowing distribution between hydroxyapatite powder (HAp) and uneven epoxy resin so that the hydroxyapatite powder (HAp) has a free group with a high specific surface area forming a weak cross bond to cover the void area arising from the presence of oxygen trapped between its constituent particles[16] [17].

- **Hardness Brinell Strength**

Hardness is the resistance of material to penetration on the surface.

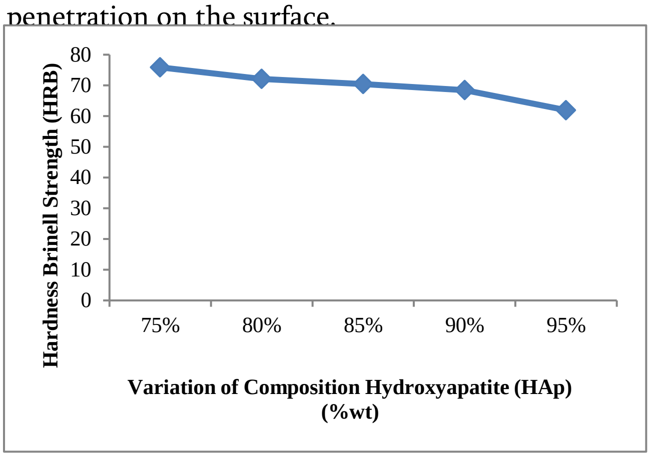


Figure 7 : Test result hardness brinell strength of bioveneer based HAp/epoxy resin on various variations composition

In Figure 7 above shows that the optimum condition obtained a high hardness value in the composition of hydroxyapatite (HAp): epoxy resin (75: 25) wt with a hardness of 75.87 HBR of which conventional dental veener hardness standards are 73-76 HBR. The hardness value decreases when the increase in the filler mass of hydroxyapatite (HAp). This is because in the manufacturing process with conventional mould techniques and cold press which allows the distribution of epoxy resin with hydroxyapatite powder is not evenly distributed so it does not achieve good inter-surface bonding between composite composers. In addition, there is a vacuum between particles that causes trapped oxygen during the compaction process and the presence of impurities[18].

D. **Characterization of Surface Morphology and Elements in HAp/Epoxy Resin**

- **Scanning Electron Miscroscope (SEM)**

SEM analysis was tested to determine the morphological form of composite particles of hydroxyapatite (HAp) from clam shell powder reinforced by epoxy resin as a dental veneer with variations in composition (75 : 25)%wt, (85: 15)%wt and (95 : 5)%wt with a magnification of 2000x.

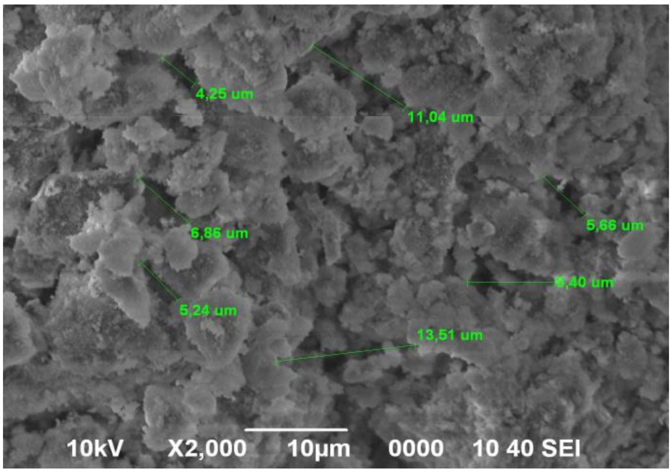


Figure 8 : Form of SEM micrographs of dental veneer surfaces based on hydroxyapatite (HAp) and epoxy resin composite with variations of composition (75: 25)%wt

Figure 8 shows that the morphology of dental veneers based on hydroxyapatite (HAp) and epoxy resin composite was the most optimum in variations of (75 : 25)%wt at a magnification of 2000x which showed small cavities with even distribution of cavities. These cavities occur due to the effect of pressure during the 300 MPa cold compaction process. The dental veneer at this optimum composition has an average pore diameter size of 7.85 µm. Furthermore, the analysis of the Energy Dispersive X-Ray Spectrometer (EDX) was carried out to determine the elements contained in the dental veneer particles through a graph of the relationship between the elemental energy (keV) parameters on the x-axis and the intensity of the second counting (cps) on the y-axis. In Figure 9 it is known that the dominant element is found in the dental veneers based on composite hydroxyapatite (HAp) powder and this epoxy resin is Ca (Calcium), O (Oxygen), and P (Phosphorus). These three elements are the main constituents of dental veneers where hydroxyapatite derived from ark clam shells is composed of compounds ($Ca_{10}(PO_4)(OH)_2$)[18][19][20]. In addition, these dental veneer samples show several impurities such as C (Carbon) and Cl (Chlorine) [19] [20].

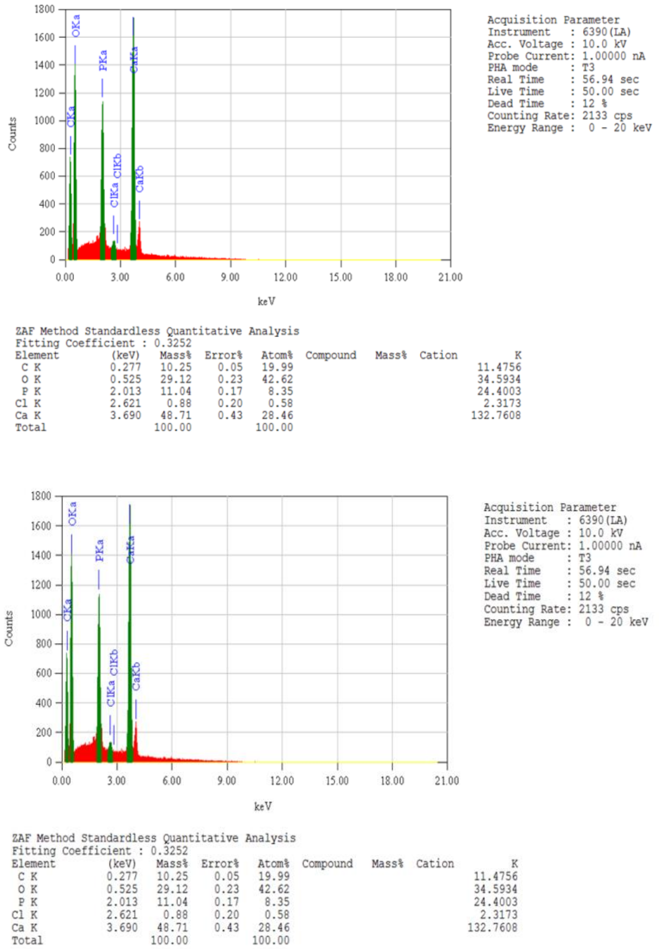


Figure 9 : The result a readings of elemental content in dental veneers in variations of composition of (75: 25)%wt with EDX

The following is a SEM analysis of dental veneers based HAp/epoxy resin composite in a low composition variation that is (95 : 5)%wt with a magnification of 1000x as follows:

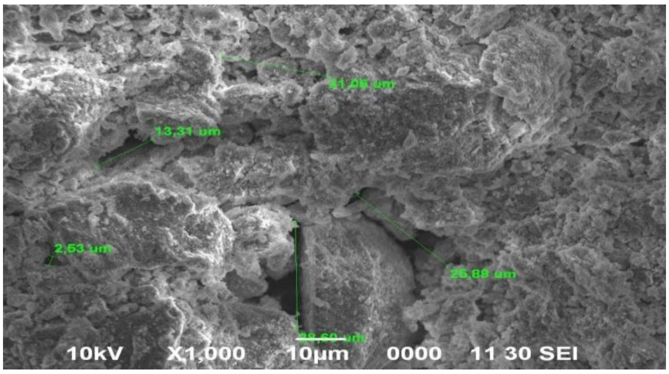
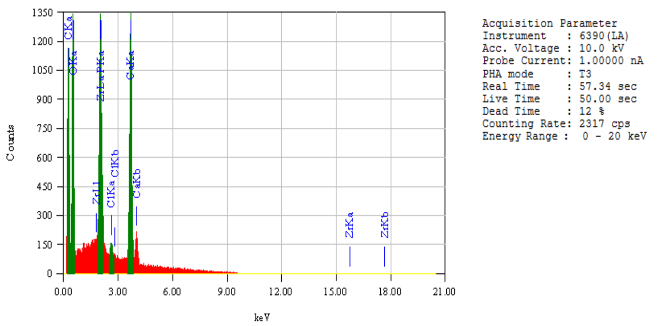


Figure 10 : Form of SEM micrographs of dental veneer surfaces based on hydroxyapatite (HAp) and epoxy resin composite with variations of composition (95: 5)% wt

*Fi*gure 8 shows that *the* morphology of dental veneers based on hydroxyapatite (HAp) and epoxy resin *composite* was the minimum variations of (95: 5)% wt.

Based on Figure 11 it is shown that there is a large cavity in which the cavity has an average cavity diameter size of 18.276 μm. With the analysis of the Energy Dispersive X-Ray Spectrometer (EDX) as



ZAF Method Standardless Quantitative Analysis
Fitting Coefficient : 0.3719

Element	(keV)	Mass%	Error%	Atom%	Compound	Mass%	Cation	K
C K	0.277	17.22	0.06	33.69				17.0271
O K	0.525	24.69	0.23	36.27				31.7984
P K	2.013	6.63	0.21	5.03				15.4961
Cl K	2.621	1.00	0.23	0.66				2.6387
Ca K	3.690	34.55	0.47	20.26				97.8433
Zr L	2.042	15.90	0.60	4.10				32.8521
Total		100.00		100.00				

Figure 11 : The result a readings of elemental content in dental veneers in variations of composition of (95 : 5)%wt with EDX

IV. CONCLUSION

In the research obtained composite materials for dental veneer applications based on hydroxyapatite powder (HAp) from ark clam shells reinforced with epoxy resin composite produced the optimum composition, namely variation of composition (75: 25)%wt has good physical properties with a density of 0.1185 x 10^3 kg/m^3, water absorption 1.163%, composed of -PO_4 (phosphate) and -CO_3 (carbonate) groups of hydroxyapatite and HOH (hydroxyl) groups of epoxy resins. Mechanical properties with a rapture strength of 116 MPa, tensile strength of 66.12 MPa, elastic modulus of 3297.99 MPa and hardness of 75.87 HBR, which results in the characteristics of conventional dental veneer standards.

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