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Improvement of Dental Composite Resin Using Supra -Nano Chicken thigh Bone Fibers

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Abstract

Supra nanoparticles (submicron) of Chicken bones fibers were used (before and after treated with citric acid solution) as additives to dental composite with the weight ratios (1%, 2% and 4%). The main mechanical tests represented by hardness, wear resistance and compression strength was carried out on the improvement dental composites.

The addition of treated supra nanoparticles Chicken thigh bones with particles size (300 nm) by weight ratio (1%) to ordinary dental composite resin , significantly improves all of its mechanical properties, in addition to the increase the value of the its glass transition temperature from 43^{o}C to 45.4 ^{o}C. The techniques X-ray diffraction (XRD), Energy Dispersive Spectroscopy (EDS) and Electron microscope images (SEM) were used to interpretation all the results that are improved mechanically and thermally.

Keywords: dental composite, epoxy, hardness, wear resistance, compressive strength, chicken thigh bone.

1. Introduction

Dental amalgam toxicity due to the presence of the element mercury in its composition, therefore dental composite resin was used instead of it, composites resin is a liquid with high viscosity, which hardens after polymerization, by applying external energy [1]. There are two phases in composite resin a matrix phase, as polymer and the reinforcing phase as fillers[2]. The oldest resin Polymetyl methacrylate (PMMA) was used in the middle of the last century, but it's use accompanies many problems such as shrinkage, which leads to lower wear resistance. To overcome the shrinkage problem the quartz was used as a filler in PPMA resin, but quartz very hard, so the composite resin was very difficult to polish [1] .

The monomer matrix currently used in composite resin is bis phenol A-glycidyl (Bis-GMA) with chemical composition ($C_{29}H_{36}O_8$), this monomer has a reactive carbon double bond at end of monomer chains that very increase during polymerization. Triethylene glycol dimethacrylate (TEGDMA) that has low molecular weight was added to (Bis-GMA) as a diluter to reduce its viscosity and control the volumetric shrinkage[4]. Various mineral fillers like aluminum silicate, zirconium and silicate zinc glasses, are used to reinforce the dental composite risen to reduce its shrinkage during the curing process [5].

To begin the polymerization and hardening process of dental composite resin, External energy like (chemical, heat and radiant) where be used to combine an electrons with the free radicals that liberated (from methacrylate monomer chains) to form covalent bonds[1]. The viscous liquid transforms to viscoelastic solid with higher density because of the shrinkage, the polymerization shrinkage reduces by increasing the filler amount and the molecular weight of the resin [5].

Bone is composite material consist 35% organic material like proteins (collagen) with water and 65% inorganic material named carbonate- hydroxy- apatite [6].

The apatite is pale green to purple mineral consists of calcium phosphate with some fluorine, chorine and some elements.The natural Calcium phosphates (CP) contain the hydroxyapatite (HA) and tri -calcium phosphate (TCP). Both (HA) and (TCP) are bio ceramics, the (TCP) has higher rate of degradation, while the (HA) was biocompatible and bioactive because it's bonding properties with the surrounding tissues [7] .

In this research, to extract the (CP) from chicken thigh bones, the citric acid solution used to remove the organic material, this may be observed by the color change, where the raw bone fibers have brown color but the color changed to white after treated with acid solution.

The dental composite was developed by adding certain percentage of supra nano Chicken thigh bone particles before and after treated with (5%wt.) of citric acid solution, the chicken thigh bone powder was environmentally friendly material and does not contain any toxicity in its chemical composition in addition to containing natural calcium which increases the resistance of the teeth to external stresses (hardness, wear, and compressive strength) that the teeth are exposed to, especially the molar (black teeth).

2.2. materials and methods

2. 2.1 Materials

The composite resin used in this work was a monomer matrix, with trade name (SHOFU BEAUTIFIL II) this matrix containing the bis phenol A-glycidyl (Bis-GMA) and Triethylene Glycol dimethacrylate (TEGDMA).
SHOFU BEAUTIFIL II contains S-PRG fillers based on fluorocarbon alumina silicate glass, polymerization initiator and pigments to decreased polymerization shrinkage.
The size range of the spherical submicron filler was (0.1- 0.3 Micrometer) with mean particle size (0.2 Micrometer), which was designed to wear resistance and to get the glossy tooth.
SHOFU BEAUTIFIL II be sensitive to LED rays with a wavelength ranging between (440-500 nm) and curing time (10 Sec) or to Halogen rays with a wavelength ranging between (400-500 nm) and curing time (20 Sec) to start the hardening process.

This technology of hardening is named a radical amplified photo polymerization initiator (RAP). The Chicken thigh bones were washed well and dried in oven at temperature (60 ^{o}C) before milling to supra- nano fibers, that have particles size ranged between (100-1000 nm), bigger than nanoscale particle size.

The chicken thigh bones fibers of size (0.3 Micrometer) and density (1.8 gm/cm^3) used as reinforcement phase addition in SHOFU BEAUTIFIL II (hybrid composites).

Some of chicken thigh bones fibers were treated with the citric acid solution (with concentration 5%) for 24 hours to get rid of organic material, then washed well with water for three times and dried in oven for 7 hours before adding to dental composite resin.

2.2.2 Method

A dental composite resin mixed with raw (not treating) and treating Chicken thigh bones fibers with the weight ratios % 1, 2% and 4%. Figure (1) shows the steps of dental resin solidification, where the resin Sitting on a epoxy specimen with rectangular dimensions ~ (28×11×11 mm). This is done first by placing a binder to the upper surface of an epoxy specimen, to connect the dental filling with epoxy material, that simulates the gums in humans.The second step is to adjust the surface of the filling well to a certain thickness and the shine ultraviolet rays (LED) on it for a period of ten seconds until it solidifies well, then a second layer of the filling is placed and rays are shed on it, in the same way as before, to ensure the solidification process for each layer and obtain a strong dental filling.

In this research, the (RAP) polymerization was done by using (LED) curing light, model: CL. N1, voltage (100 -240 V), frequency (50-60 Hz) and intensity (260 mw/cm^2).

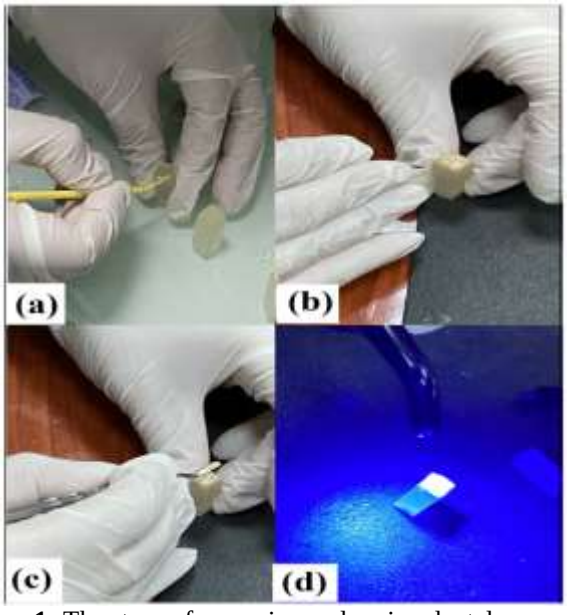

Figure 1: The steps of preparing and curing dental composite.

a) Putting the chemical bond on the surface of the epoxy.
b) Laying the dental composite on upper surface of epoxy.
c) Adjusting the dental composite on epoxy.
d) Hardening of the dental composite by UV- light.

2.3.Mechanical tests

2.3.1 Hardness test

Shore (D) hardness device model TH210 manufactured in Italy was used to check the hardness values of ordinary composite resin and the improved dental composite resin by adding different weight ratios of untreated and treated the supra nano Chicken thigh bones particles with citric acid solution.

2.3.2 Wear test

The wear rate of ordinary and improved dental composite was done using pin-on-disk, the load applied on the specimen was (500 gm).

The specimen under the wear test was rotated around the center of the metallic disk by a distance (r = 9 cm) for time (t = 5 min) and the number of turns was (n = 375 cycle/min).

Equation (1) used to is determine the wear rate [8].

$$\text{Wear rate} = \frac{Wo - W}{s} \quad \ldots\ldots.(1)$$

Where

W_o: the initial weight specimen.

W: the final weight specimen.

The value of sliding distance (s) determine using equation (2).

$$s = 2лrnt \quad \ldots\ldots. (2)$$

2.3.3 Compressive test

The test was done on specimens of a dental composite resin holding on the epoxy using a universal compression test machine, the compressive stress was calculated using the equation(3) [8].

$$\text{Stress} = \frac{F(N)}{A(mm^2)} \quad \ldots\ldots..(3)$$

Where

F: is the compressive force applied to the specimen.

A: is the base area.

2.4 Differential scanning calorimetry (DSC)

DSC (separate EVO 131) was used to determine the glass transition temperature of ordinary and reinforced dental composite resin, with heating rate (10 ^{o}C/ min) and temperature range (20-400 ^{o}C). DSC measure the quantitative heat flow as a function of the temperature of sample or time, for knowing how a substance behave towards an increase temperature, this curve provides the information on softening temperature Tg, which the resin transform from a hard (glassy) to viscous liquid [12] .

2.5 X-ray diffraction technique (XRD)

X-ray diffraction device of the type (SHIMADZU Japan) (XRD600) has been employed using the X-Ray with wavelength (1.54 A^o), the scanning range of the rays ranging between (10- 80 degrees).

2.6 Energy Dispersive Spectroscopy (EDS)

The elements compositions of ordinary and reinforced dental composite resin were fixed using (EDS) technique (Procar) made in Germany, equipped with the SEM technique.

2.7 Scanning electron microscopy (SEM)

To examine the dental filling samples and determine the homogeneity of the filling in these samples, they were coated with a thin layer of gold to be photographed in the electronic scanning device (Tescan) manufactured in England.

3. Result

3.1 Hardness test

The hardness values for the ordinary and the reinforced dental composites resins, are shown in figure(2). Hardness values are increased from (95) for the ordinary dental composite resin to highest values (96) and (97.05) by adding weight ratio (1%) of raw and treating Chicken thigh bones fibers, respectively .

That means the hardness value of ordinary dental composite resin, was improved by adding the chicken thigh bones particles, causes the improvement in the hardness values, this results are agreed with [10].

It is possible to observe that the adding the chicken thigh bones particles with a small proportion made these natural fibers well dispersed in ordinary composite resin, which helps to strengthening the composite resin matrix by blocking the propagation of the crack [10].

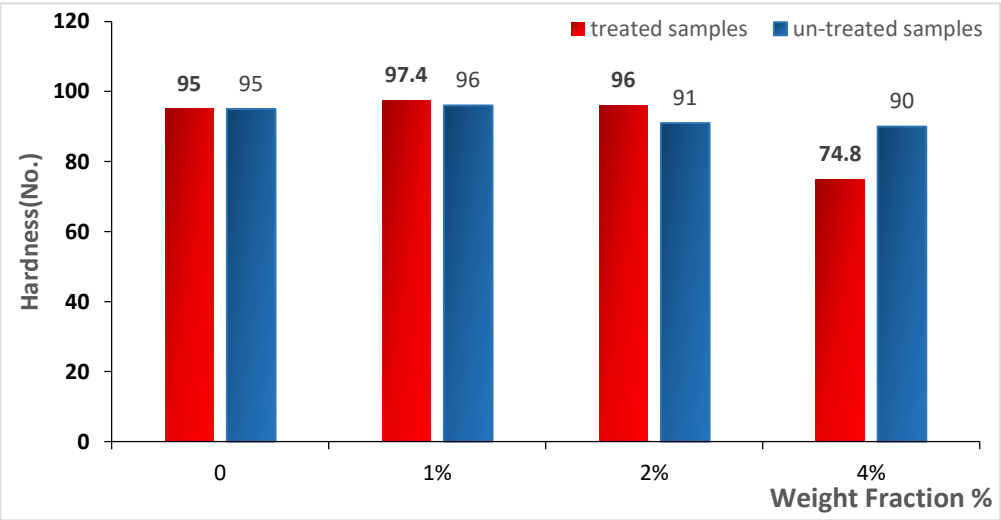

Figure (2): the effect of adding the raw and treating Chicken thigh bones fibers in different weight fractions on the hardness value of dental composite.

3.2 Wear test

To determine the wear rate of a dental composite that simulates the process of chewing food in opposite directions by the molar or posterior teeth, a wear adhesive test was performed on the ordinary and reinforced dental composite.

Figure(3) shows that the ordinary composite has a high value of wear rate (4.25×10^{-9} gm/cm), compared to the lower rate values (2.83×10^{-9} gm/cm) and (1.42×10^{-9} gm/cm) of improved dental composite resin, that reinforced with chicken thigh fibers with weight ratio (1%) before and after treated with acid solution.

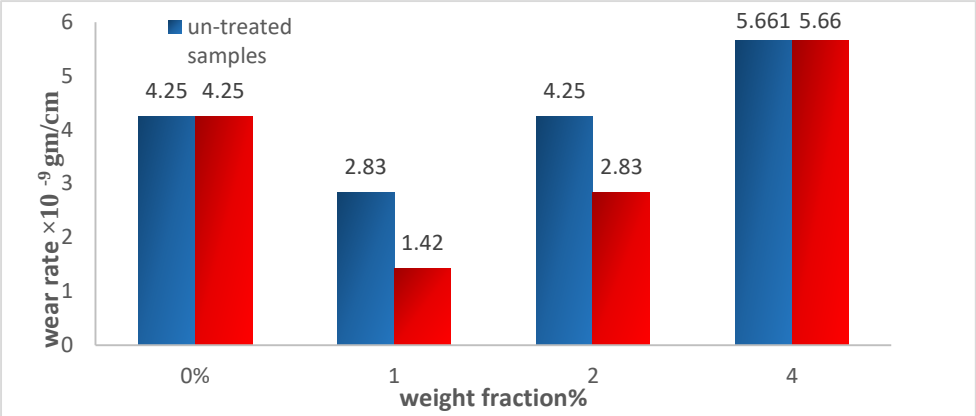

Figure (3): wear rate of ordinary and reinforced dental composites.

The reducing in wear rate in improved dental composite resins, means the (1% wt.) of chicken thigh fibers are considered an important factor in increasing the wear resistance of the dental composite and reducing the effect of the process of chewing food (opposite stresses on the dental composite).

By comparing the hardness values and the wear rate values in the two figures (2) and (3), it appears that an inverse relationship between the hardness values and the wear rate of the material, this completely agrees with [8,9].

3.3 compression test

In this test, an axial load was applied to the specimens.The samples of epoxy that holding the ordinary and improved dental composite resin with adding the chicken fibers by the weight ratios (1%, 2% and 4%) before and after treating, were subjected to compressive forces, as summarized in table (1), the values of change length ($L - L_o$) and stress of the specimens are increased with the increase in the values of the applied force.

Table 1: compressive strength of ordinary and reinforced dental composite resin holding on epoxy bars.

Ordinary dental composite				Ordinary dental composite +1% C.B				Ordinary dental composite +2% C.B				Ordinary dental composite +4% C.B			
f (N)	Δ L (mm)	(σ) (N/mm2)	ε	F(N)	Δ L (mm)	(σ) (N/mm2)	ε	F(N)	Δ L (mm)	(σ) (N/mm2)	ε	F(N)\	Δ L (mm)	(σ) (N/mm2)	ε
0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
500	0.1	6.2703	0.004	500	0.1	6.384	0.004	500	0.1	6.31	0.0043	500	0.1	6.377	0.003
1000	0.21	12.54	0.008	1000	0.4	12.768	0.005	1000	0.23	12.626	0.009	1000	0.33	12.755	0.013
1500	0.45	18.81	0.018	1500	0.63	19.152	0.008	1500	0.66	18.939	0.025	1500	0.5	19.133	0.019
2000	0.93	25.08	0.037	2000	0.81	25.536	0.01	2000	0.91	25.252	0.035	2000	0.8	25.51	0.031
2500	1.2	31.35	0.048	2500	1.3	31.92	0.016	2500	1.4	31.565	0.054				
				2650	1.5	33.835	0.059								

Epoxy				Ordinary dental composite +1% TC.B				Ordinary dental composite +2% TC.B				Ordinary dental composite +4% TC.B			
f (N)\0	Δ L (mm)\0%	(σ).	ε	f (N)	Δ L (mm)\1%	(σ) (N/mm2)	ε	f(N)	Δ L (mm)\2%	(σ) (N/mm2)	ε	F(N)	Δ L (mm)	(σ) (N/mm2)	ε
0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
500	0.05	6.4355	0.002	500	0.1	6.341	0.0041	500	0.13	6.47	0.0052	500	0.12	6.12	0.0047
1000	1.4	12.8711	0.057	1000	0.5	12.683	0.02	1000	0.53	12.94	0.021	1000	0.43	12.25	0.016
1500	2.1	19.5025	0.085	1500	0.91	19.025	0.037	1500	0.71	19.41	0.028	1500	0.81	18.37	0.031
1650	2.9	21.2382	0.117	2000	1.29	25.367	0.053	2000	1.01	25.88	0.04	2000	1.17	24.5	0.045
				2500	1.57	31.709	0.064	2500	1.41	32.36	0.057	2200	1.31	26.95	0.051
				3000	2.17	38.05	0.089	2650	1.7	34.3	0.068				

Figure (4) shows the relationship between compressive strength of samples and weight ratios of chicken thigh bones fibers in dental composite resin, where the weight ratio (1%) was the best proportion of chicken thigh bones fibers addition.

The treatment of chicken thigh bones fibers with citric acid solution will improve the compressive strength of sample from (33.835 Mpa) for improving the dental composite with raw chicken thigh bones fibers to (38.05 Mpa) for improving the dental composite with treatment chicken thigh bones fibers, at the best amount value of powder addition (1%), this because the get rid of organic material that exists in raw chicken thigh bones fibers.

Figure (5) shows that the weight ratio (1%) of Chicken thigh bone fibers improved the strain of dental composite resin , from (0.048) for ordinary dental composite to (0.059) for dental composite with raw chicken bones fibers and to (0.089) for dental composite resin with treated chicken bones fibers, that mean the (1% of treated chicken bone fibers) was enough to overcome the shrinkage problem in dental composite resin.

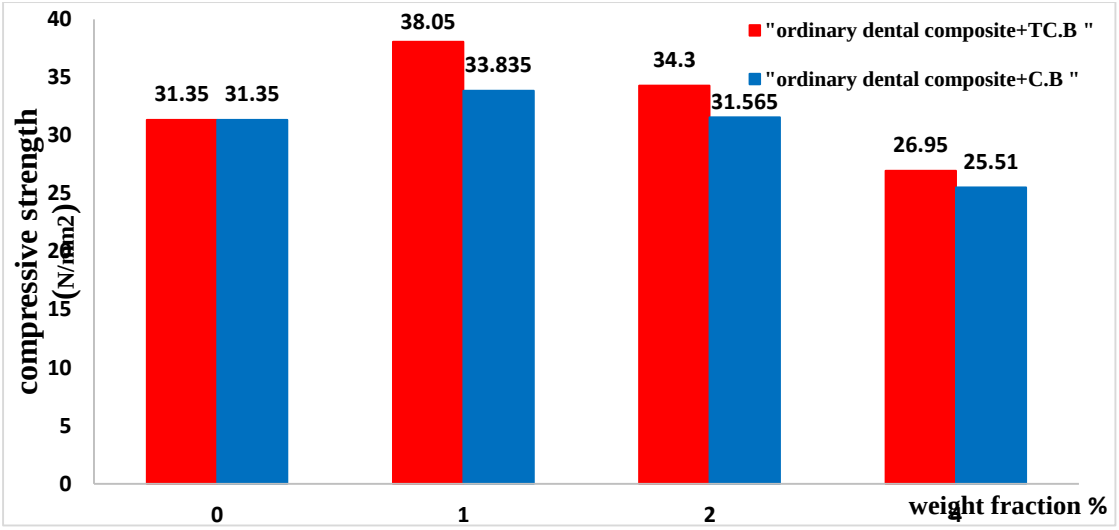

Figure4: compressive strength values of samples with different weight fractions of fibers before and after treated.

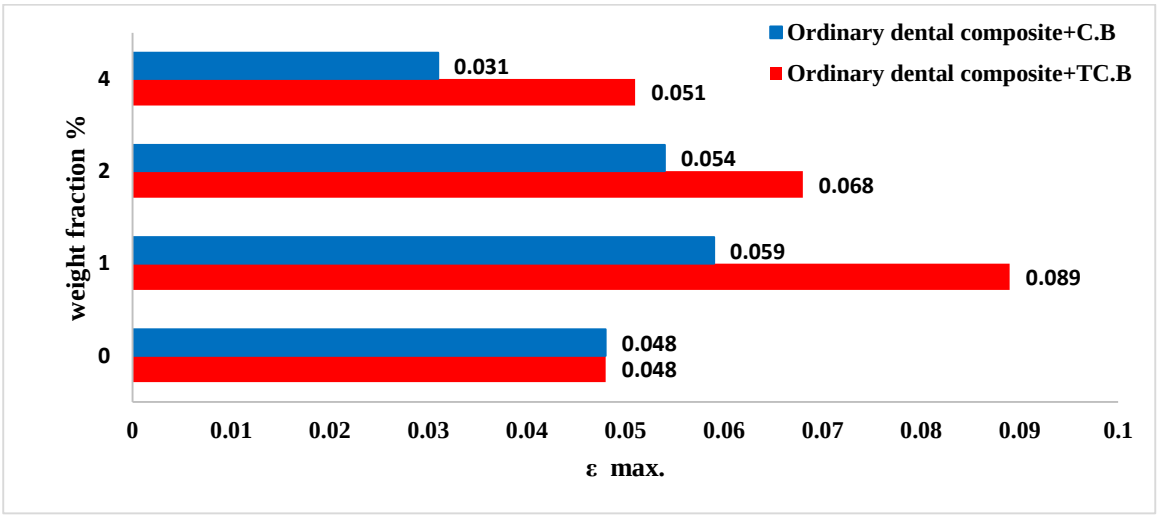

Figure5: the maximum values of samples strain with different weight fractions of fibers before and after treated.

Table (1) shows that the compression strength of pure epoxy was (21.23 Mpa) by applied the force (1650 N), the compressive strength of sample (epoxy + dental composite) reach to maximum value (31.35 Mpa) by applied the force (2500 N). The compressive strength will increase to (33.835 Mpa) by applied the force (2650 N) for the sample (epoxy + dental composite/ 1 % untreated chicken fibers) and to (38.05 Mpa) by applied the force (3000 N) for the sample (epoxy + dental composite/ 1 % treated chicken fibers), that mean the net compressive strength are (10.12 Mpa), (12.6 Mpa) and (16.8Mpa) for ordinary dental composite resin, dental composite resin mixed with 1% raw chicken bone fibers and dental composite resin mixed with 1% treated chicken bone fibers, respectively, these results are agreed with [5].

3.4 Differential scanning calorimetry (DSC)

Figure (6) shows that the ordinary dental composite have the glass transition temperature (43 ^{o}C) which is agree with the range of glass transition values in the literature [13].

By the addition the (1%wt.) of treaded supra nano Chicken thigh bone to this dental composite, the glass transition temperature was increase to (~ 45.5 ^{o}C) that mean the improvement in glass

transition temperature by (~ 6%) , but with adding the ratio (1%) of untreated chicken thigh bones fibers to dental composite was not affected on its glass transition value , this because the values of the glass transition temperature is affected by the percentage of elements composition in the fillers [12] .

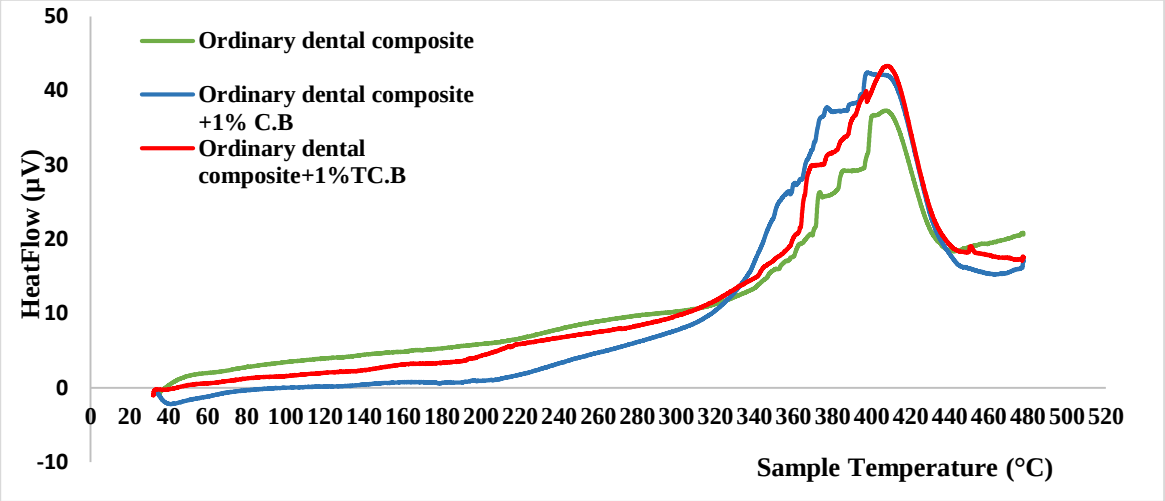

Figure 6 : Differential scanning calorimetry curves of ordinary and improvement dental composites.

3.5 X-ray diffraction Figure (7) shows the X-Ray diffraction patterns of raw (un treated) and treat chicken thigh bone fibers, because the eliminating of organic material, the X-ray diffraction of treated chicken bone fibers have distinctive peaks and more crystalline structure compared with the peaks of raw bones fibers .

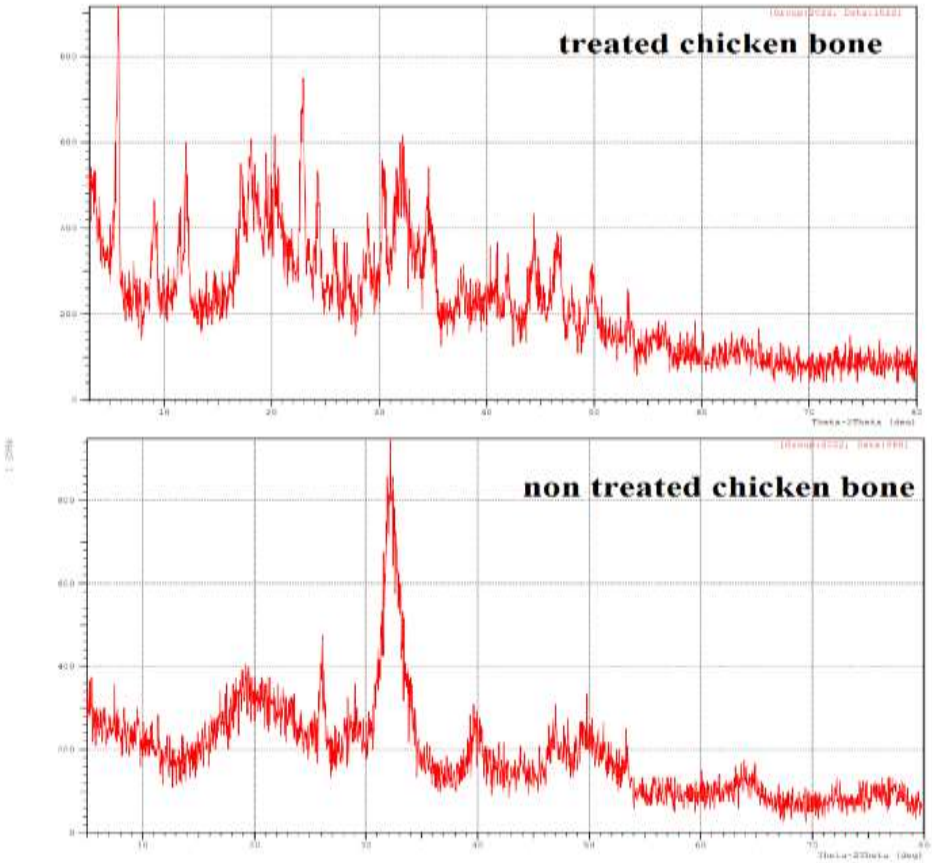

Figure 7 : X-ray diffraction patterns of raw and treated chicken bones fibers.

The Intensity of three mains peaks, diffraction angles and the full width at half maximum(FWHM) of raw and treated chicken bones fibers, were summarized in table (2).

Using Scherer formula (4) and and the informations in table (2) the crystallites size (D) of raw and treat chicken thigh bone fibers were estimate.

crystallite size (D) of raw chicken thigh bone fibers was about (26 nm) and increase up to (44.77 nm) for treating Chickenbones fibers, this results areagreed with the crystallite size (38.48 nm) of heat treatment chicken bone fibers, where the bones fibers calcined at 1000^{o}C [7].

Table 2 : X-ray diffraction angles and the (FWHM) for raw and treated chicken bones fibers

	Peak no.	θ (deg)	FWHM(deg)	Intensity (counts)
Raw chicken bone fiber	**11**	**32.3126**	**1.744**	**101**
	12	**33.9589**	**1.0134**	**27**
	16	**18.9063**	**2.16**	**26**
Treated chicken bone fiber	**4**	**5.8191**	**0.325**	**94**
	32	**22.8787**	**0.4733**	**73**
	14	**12.1389**	**0.406**	**52**

$D=\frac{K\ \lambda}{B\cos\theta}$ ... ... (4)[7]

Where

λ: X ray wave length .

K : Scherrer constant , which equal to 0.94

θ: is the diffraction angle

B: is full width at half maximum

3.6 EDX measurements

Figures (8-10) and table (3) shows the EDS peaks and chemical compositions of (dental composite resin), (dental composite resin /1% raw C.B Fs) and (dental composite resin/1% treated C.B Fs) respectively .

The elements C and O are consistent in ordinary dental composite with high rate, some elements like N, Si, F and AL were found in small percentages.

By adding the raw chicken thigh bones fibers to dental composite resin, the structure of final composite will changed, some elements like Ca and W will appear in high rate (31.47%) and (11.18) , other elements like Na and Mn were appeared in small percentages , while the percentages of O, C , Si and Al were changed.

The percentage of O element in raw chicken thigh bone fibers in table (3) was very high, this because the chicken bone marrow continued stem cells that can be grow to red cells, which carries oxygen to every parts of the body , this results are agreed with [14] .

The treatment of chicken thigh bones fibers will be greatly increased the Ca and C elements in the structure of improvement dental composite resin, this may the reason of the improvement of (dental composite resin / 1% treated chicken bone fibers) in different mechanical properties and in the increase its glass transition temperature .

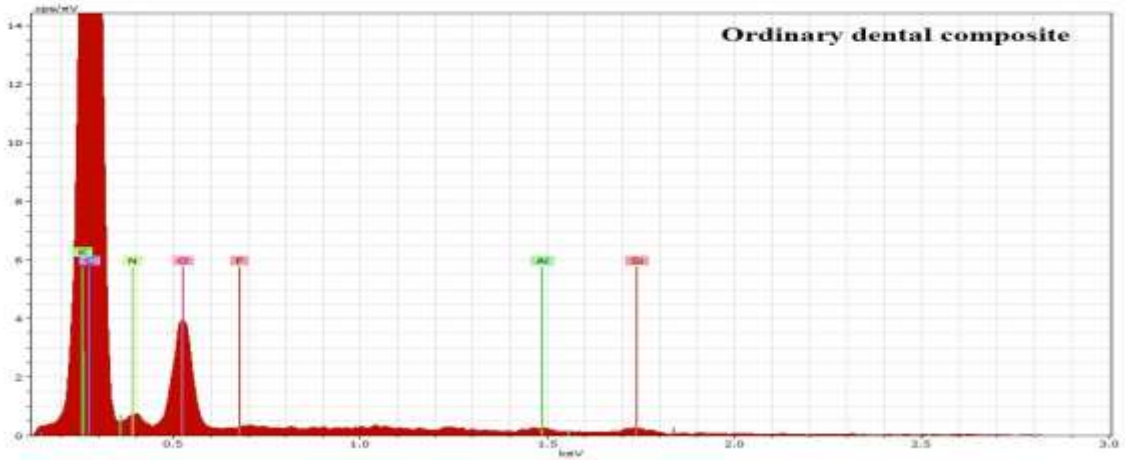

Figure 8: EDX pattern of ordinary dental composite resin

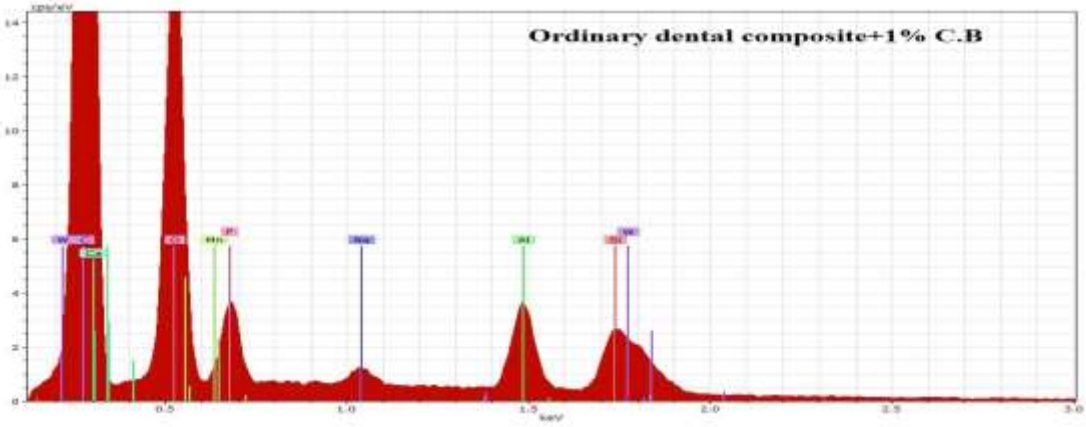

Figure 9: EDX pattern of ordinary dental composite resin with 1% raw C.B. $F_{s.}$

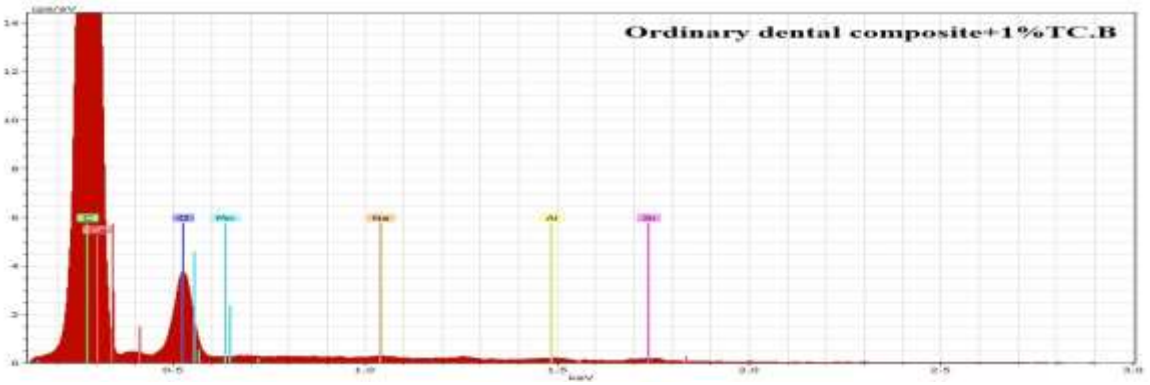

Figure 10: EDX pattern of ordinary dental composite resin with 1% T.C.B .$F_{s.}$

Table 3: the elements compositions of ordinary and improvement dental composites.

Ordinary dental composite		**Ordinary dental composite +1% C.B.**		**Ordinary dental composite +1% T.C.B**	
Element	**Wt. %**	**Element**	**Wt. %**	**Element**	**Wt. %**
C	75.97	Ca	31.47	Ca	62.79
O	17.02	O	23.71	C	27.8
N	4.54	C	14.86	O	7.66
Si	1.77	W	11.18	Mn	0.94
F	0.40	Si	7.29	Si	0.59
Al	0.30	Al	5.63	Al	0.19
K	0.00	F	4.85	Na	0.03
		Na	0.77		
		Mn	0.23		

3.7 Scanning electron microscopy SEM

Figure (11) shows the morphology of ordinary dental composite, with different magnification, a high degree of roughness with white color in Images (a) was appeared, this due to agglomeration of the fillers like silicate in this resin , which causes crack initiation , and low in mechanical properties like hardness, wear, the weakness in its compressive strength and low in compressive strain and glass transition temperature values, causes the shrinkage of composite.

Images (b) shows some gaps and the surface was more fragile if they compared with images (c), this because the presence of oxygen element in high rate in the chemical composition of raw chicken thigh bones fibers.

The adding of the Chicken thigh bones fibers with weight ratio (1%) to dental composite, lead to improve its hardness and wear resistance, this because of the nature of bio ceramic filler (chickenbone fibers), which play a good roll in strengthening the dental composite resin With adding the treatment Chicken thigh bones fibers to dental composite resin with weight ratio (1%) the mechanical properties were more improved , these improvements due to two reasons, the first was the abundance of calcium in high rate, that leads to strengthening the dental composite against the external stresses , the second reason was the good dispersion of this suitable value of addition in dental composite matrix, which lead to good in interfacial bonding between the fillers and dental composite resin , as appear in microstructure photographs in figure (11 c).

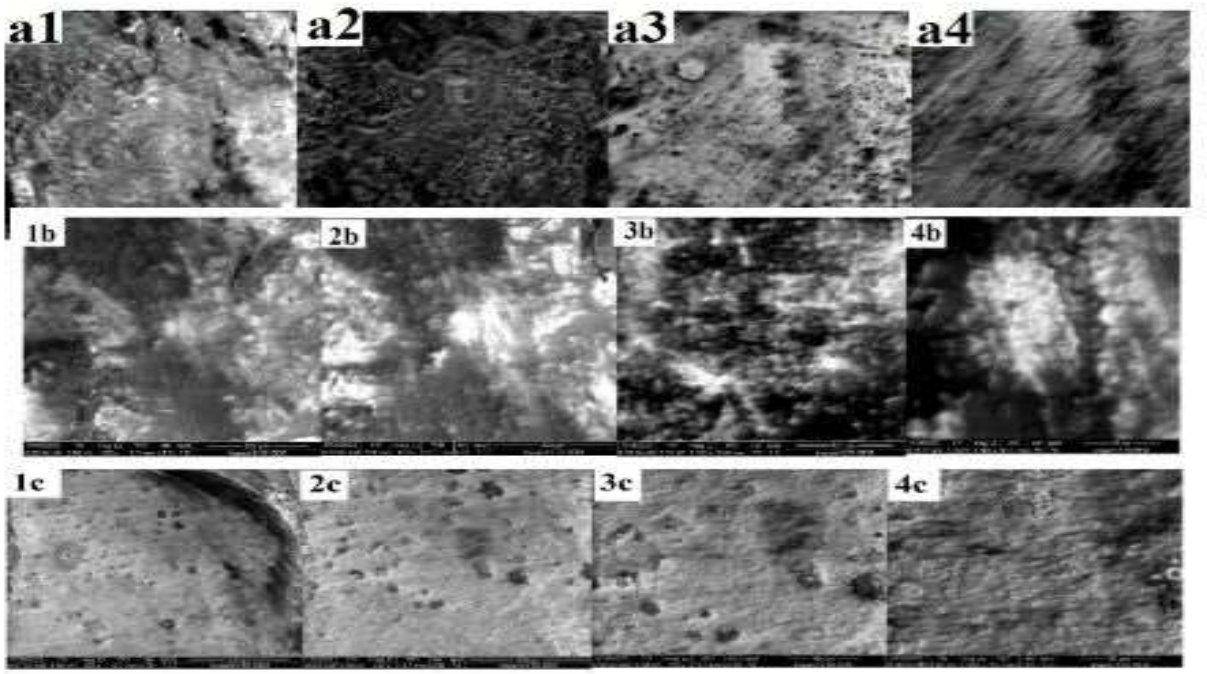

Figure 11: SEM images of fracture surface of a) dental composite.

b) Dental composite resin/ 1% C.BFs c) dental composite resin / 1% T .C.BFs with magnification 1) 200 × 2) 400× 3)1000× 4) 1500×

Conclusions

Chicken thigh bones fibers were selected as natural bio-ceramic particles to improve the mechanical properties of dental composite resin by incorporated this raw bone fibers. To get further improvements in mechanical properties of dental composite resin like hardness values, wear resistance and compressive strength, a treat chicken thigh bones fibers was used in small weight percentage (1%) .

The treatment of chicken thigh bones with a citric acid solution, considered as economical method for calcium phosphate extraction that leads get the improvement in mechanical properties of dental composite, by (2.52%), (66.58%) and (21.37%) for hardness, wear resistance and compressive strength respectively, also a big improvement achieved in the maximum compressive strain (85.41%) which means the overcome of shrinkage problem in dental composite resin.

The addition of treated chicken thigh bones fibers by 1% of weight ratio to dental composite cases the raising in its glass transition temperature which rises by the ratio (~ 6 %), also the (1%) was optimal weight ratio of addition to improve the crystallinity of dental composite resin and

changing its elements composition by fortify it with natural calcium which increases the its resistance to external stresses .

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