Effect of resin composite composition, shade and curing system on fracture toughness

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Abstract: Objectives: Evaluation of the effect of resin composite composition, shade and curing system on fracture toughness. **Methods:** A total of 40 nanohybrid resin composite specimens; 20 of each resin composite type; 10 of each shade, were prepared. One of the two nanohybrid resin composites was ormocer-based resin composite (Ceram X) and the other one was di-functional methacrylates-based resin composite (Artiste). Light and dark shades (A1 and A3.5) of each material were chosen. Half of these specimens (20 specimens) were activated by halogen light curing unit and the other half was activated by light emitting diode (LED) light curing unit. The fracture toughness values were determined by the universal testing machine using the Single Edge Notched Beam (SENB) specimens. **Results:** The three-way (ANOVA) test revealed that the ormocer-based resin composite (Ceram X) showed significantly higher fracture toughness values (2.61±0.2 MPa.m$^{1/2}$) than the di-functional methacrylates-based resin composite (Artiste) (2.36 ±0.3 MPa.m$^{1/2}$). Furthermore, the lighter shade (A1) showed significantly higher fracture toughness values (2.59 ±0.3 MPa.m$^{1/2}$) than the darker shade (A3.5) (2.38 ±0.3 MPa.m$^{1/2}$) for both resin composite types. However, there was no significant difference between the fracture toughness values of both types of resin composites when cured with either LED or halogen light curing system. **Significance:** Ormocer-based nanohybrid resin composite restorative material is a strong treatment option for stress bearing areas. Moreover, lighter shade of resin composites demonstrates better fracture toughness than its darker one of the same brand; this should be considered during curing.

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Keywords: *Resin composite; Nanohybrid; Difunctional methacrylates; Ormocer; Fracture toughness; LED; Halogen; Light curing unit*

1. Introduction:

Resin composites represent a class of materials widely used in restorative dentistry because of patient demands for better aesthetics. These encourage manufacturers to improve the resin composite chemistry together with the advancement of light curing systems.

Nanotechnology provides resin composite with filler particles that are dramatically small and can be impregnated at high concentrations in the resin matrix. This will enhance the mechanical properties, as well as, the aesthetic requirements of resin composite restorations, Moszner and Salz(2001); Muselmann (2003). Ormocer is an acronym for organically modified ceramics. They represent another new technology based on solution and gelation processes using particles comprising silicones, organic polymers and ceramic glasses that is embedded in the resin matrix, Fraunhofer (2009). The combination of ormocer resin composite technology and nano-particle fillers has been developed in an attempt to produce resin composites of better mechanical and aesthetic durability, Ceram (2003); Tagtekin et al., (2004); Norbert et al., (2008) .

Shading of resin composite materials is achieved by the addition of minute amounts of inorganic metal oxide pigments. Shades can range from very white bleaching shades to yellow or gray, Craig and Power (2002).Upon curing of resin composite, the curing light will be absorbed by the photoinitiators and scattered by the filler particles
and pigments. Thus, reducing the irradiance and curing effectiveness, Kawaguchi et al., (1994); Watts and Cash (1994). The effect of resin composite shade on the curing efficiency and mechanical properties has been studied. However, controversial results were obtained.

The LED curing unites have been introduced to overcome the drawbacks of the conventional halogen light curing unites. The drawbacks include: heat generation, the need for ventilating fan, limited lifetime of the bulb, as well as, degradation of the reflector and filter over time, Caughman et al., (1995); Althoff and Hartung (2000).

Fracture toughness is an important property to characterize the fracture behaviour of resin composites. It provides a valuable information about the population of flaws having the potential to cause failure. Fracture toughness (K_{Ic}) is a measure of the stress intensity or the critical stress intensity at the tip of a crack or flaw from which a crack propagates throughout a material in an unstable manner ,Ritter

(1995); Fujishimaand Ferracane (1996); Bona et al. ,(2003). Occurrence of flaws is inevitable in the processing, fabrication, or service of a material component. Flaws may appear as cracks, voids and design discontinuities. Catastrophic crack propagation of flaws within the resin composite structure can lead to marginal fracture or surface degradation and eventually failure of the restoration, Scherreret al., (2000); Rodrigues et al., (2008) .

Limited studies have been done to assess the effect of resin composite chemistry, shade and the type of curing light technology on the fracture toughness. Therefore, the present study was carried out in an attempt to evaluate the effect of resin composite composition, shade and curing system on its fracture toughness.

2. Materials and methods:

2.1. Materials:

2.1.1. brands :

Two brands of commercially available nanohybrid light-activated resin composites were used in this study. One was an ormocer-based resin composite (Ceram X) and the other was di-functional methacrylates-based resin composite (Artiste). Two different shades were selected in this study A1 as light shade and A3.5 as dark shade. The materials used, their batch number and their composition are listed in table (1).

Table (1): Details of the two resin composites tested in this study

Material	Ceram X TM Mono	Artiste® Nano Composite
Type	Nanohybrid	Nanohybrid
Shades	M1=A1 and M6=A3.5 (body)	A1 and A3.5(body)
Lot No.	0804001516	166034
Filler Type	Barium alumino-borosilicate glass,Organically modified silicon dioxide nanofillers	Barium boro-silicate glass , nano-particulated silica, zirconium silicate
Filler Content	57 % by volume,76 % by weight	66% by volume ,75% by weight
Filler size	Glass filler mean size1.1-1.5 µm Nanofillers 2.3-10 nm	Glass filler mean size0.7-1.0µm Nanofillers about 20 nm
Resin Phase	Methacrylate modified polysiloxane,conventional dimethacrylate resin	A mixture of difunctional methacrylates of PCBisGMA, BisGMA, UDMA and HDDMA
Recommended curing time /2 mm	20 seconds	10 seconds
Photoinitiator	Camphoroquinone (CQ)	Camphoroquinone (CQ)
Manufacturer	Dentsplay DeTrey,Konstanz,Germany	Pentron Clinical Technologies,USA

2.1.2. Resin composite specimens:

A total of 40 resin composite specimens have been prepared and divided into two main groups (n=20) according to the two types of resin composite used (Ceram X and Artiste). Each group was subdivided into two subgroups (n=10) representing the selected shades (A1 and A3.5).The subgroups were further subdivided into two sub-subgroups (n=5) to be cured with the different curing systems.

Two light curing systems were used for the activation of tested resin composite filling materials. Halogen light curing unit (Coltolux 75) and LED light curing unit (LEDition) were used. The technical data of the devices used are listed in table (2).

2.2. Methods:

2.2.1. Specimens preparation:

Forty Rectangular notched specimens (25 ± 2mm) x (5 ± 0.1mm) x (2.5 ± 0.1mm) were prepared in split mould held in a metallic frame with a sharp V-shaped extension providing pre-notched specimens, ASTM Designation (1997) as shown in figure (1). The uncured materials were packed in the mould, covered by Mylar strips and topped by glass plates from both sides. Pressure was applied in order to extrude excess materials and produce a flat surface.

Curing was done by dividing the specimen into thirds. The middle third of each individual specimen was initially cured according to manufacturer’s instructions for each resin composite type, where the di-functional methacrylates-based resin composite (Artiste) was cured for 10 seconds and the ormocer-based resin composite (Ceram X) was cured for 20 second. Both lateral thirds were exposed to the curing light alternately for the same curing time regime on both sides of the specimen. After curing, each specimen was carefully removed from the mould and visually inspected for voids or any noticeable defects especially around the notched area. The specimens were then stored in distilled water at 37±1°C for 24 hours in the incubator (ELYE-3 incubator, China) before testing.

Table (2): Technical data of the two light curing system

Light curing system	COLTOLUX® 75 COLOR TALK	LEDition
Types	Quartz Tungsten Halogen , high power	Light Emitting Diode , second generation
Manufacturer	Coltene/Whaledent	Ivoclar Vivadent AG
Manufacturer code	C59-0968	602108
Wavelength range	430 – 505 nm	430 – 490 nm
Light source	75 watts	3 watts
Light intensity	700 up to 1000 mW/cm^2	700 up to 900 mW/cm^2
Light guide	8 mm	10 mm

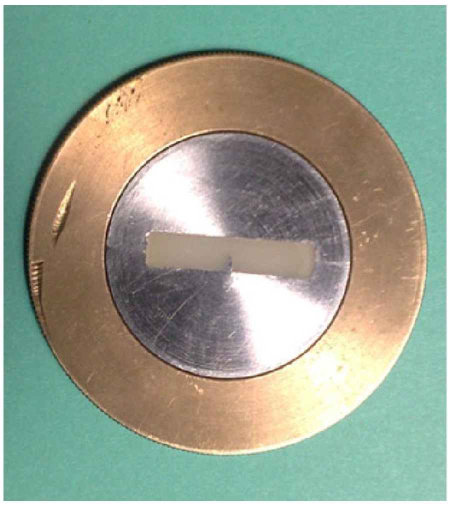

Figure (1): Split metallic mold filled with resin composite

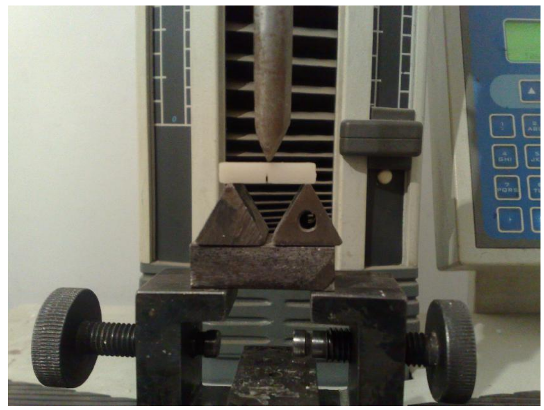

Figure (2): Fracture toughness specimen mounted under three-point bending in universal testing machine

2.2.2. Testing methods:

The specimens were tested under three-point bending using a universal testing machine (Lloyd's Instruments Ltd, UK). Each specimen was supported on two parallel stainless steel rods (10 mm in diameter) located 20 mm apart .The load was applied through a cylindrical stainless steel rod (10 mm in diameter) at the middle of the specimen as shown in figure (2). The load was applied at a cross head speed of 0.75 mm/min until fracture. Both the load and the deflection were obtained from the load-deflection curve produced by NEXTGEN software program of the computer connected to the testing machine.

2.2.3. Calculation of the fracture toughness (FT):

The fracture toughness value of each specimen was calculated from the following equation, ASTM Designation(1997).

$$\mathbf{K_{Ic} = [3\,P\,L\,a^{1/2} / 2\,b\,w^2] \times f(a/w)}$$

Where:

K_{Ic} = Fracture toughness Mode I (MNm$^{-1.5}$)
P = Load at fracture in Newton (N)
L = Distance between the support in mm (20 mm)
A = Crack length in mm = W/2 (2.5 mm)
b = Thickness of specimen in mm (2.5 mm)
w = Width of specimen in mm (5 mm)

$f(a/w) = [1.93 - 3.07(a/w) + 14.53(a/w)^2 - 25.11(a/w)^3 + 25.80(a/w)^4]$

The value f (a/w) was obtained from ASTM slandered, ASTM Designation(1997).The specimen geometry is shown in figure (3) .

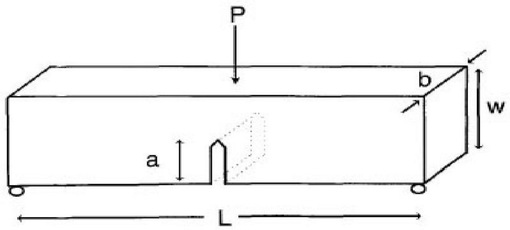

Figure (3): Specimen geometry for the determination of fracture toughness by single-edge notched method Fujishima and Ferracane (1996)

2.2.3.Statistical analysis:

Three-way (ANOVA) test with statistical package for scientific studies (SPSS 16.0) was used.

3. Results:

Regression analysis results of the effect of resin composite chemistry, shades, light curing units and their interactions on the mean fracture toughness are shown in table (3).The results showed that the selected resin composite chemistry and shades had a significant effect on the mean fracture toughness values. However, the light curing units and the interaction between all variables had insignificant effect.

Table (3): Results of regression analysis of the effect of resin composite chemistry, shades, light curing units and their interactions on the mean fracture toughness

Source of variation	Sum of Squares	df	Mean Square	*P*-value
Corrected model	1.592	7	0.227	**0.002***
Resin composite chemistry	0.583	1	0.583	**0.002***
Resin composite shade	0.448	1	0.448	**0.006***
Light curing unit	0.021	1	0.021	**0.525**
Resin composite chemistry x Resin composite shade x Light curing unit	0.118	1	0.118	**0.142**

*: Significant at $P \leq 0.05$, df: degrees of freedom, R Squared = 0.489 (Adjusted R Square = 0.377)

Regarding to the effect of resin composite chemistry on the fracture toughness values, the three-way (ANOVA) test revealed that the ormocer-based resin composite (Ceram X) had significantly higher values than the difunctional methacrylate based (Artiste) resin composite as presented in table (4).

Table (4): The effect of the resin composite chemistry on the fracture toughness of the tested resin composites.

Artiste		Ceram X		*P*-value
Mean ($MPa.m^{1/2}$)	SD	Mean ($MPa.m^{1/2}$)	SD	
2.36	0.3	2.61	0.2	**0.002***

**: Significant at $P \leq 0.05$*

On the other hand, the lighter shade (A1) possessed a significantly higher mean fracture toughness values than that of the darker shade (A3.5) of both resin composites as shown in table (5).

Table (5): The effect of the shades on the fracture toughness of the tested resin composites.

A1		A3.5		*P*-value
Mean ($MPa.m^{1/2}$)	SD	Mean ($MPa.m^{1/2}$)	SD	
2.59	0.3	2.38	0.3	**0.006***

**: Significant at $P \leq 0.05$*

Furthermore, there was no significant difference between the mean fracture toughness values of both resin composites when activated with either LED or halogen light curing units as presented in table (6).

Table (6): The effect of the light curing units on the fracture toughness of the tested resin composites.

LED		Halogen		*P*-value
Mean ($MPa.m^{1/2}$)	SD	Mean ($MPa.m^{1/2}$)	SD	
2.51	0.3	2.46	0.3	**0.525**

Table (7): The effect of the different variables' interactions on the fracture toughness of the tested resin composites.

Light curing unit	Resin composite chemistry	Resin composite shade	Mean ($MPa.m^{1/2}$)	SD	*P*-value
LED	Artiste	A1	2.58	0.3	**0.142**
		A3.5	2.38	0.5	
	Ceram X	A1	2.60	0.07	
		A3.5	2.47	0.1	
Halogen	Artiste	A1	2.28	0.1	
		A3.5	2.21	0.3	
	Ceram X	A1	2.89	0.07	
		A3.5	2.46	0.2	

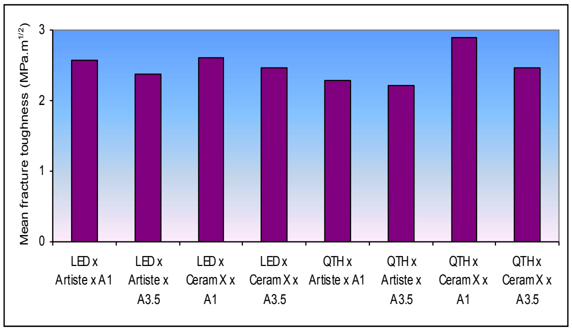

Figure (4): Fracture toughness of the different variables' interactions

4. Discussion:

Aesthetic dentistry continues to develop through innovations in resin composite chemistry and light curing systems. Such advances have markedly increased the opportunities of providing solutions to many restorative and aesthetic challenges faced by clinicians. Therefore, the utilization of the resin composite materials to restore both anterior and posterior teeth has been widely accepted.

The fracture behaviour of resin composites is an important factor that affects the longevity of the restorations in patient mouth. Fracture toughness is a mechanical property that measures the ability of the material to resist fracture by crake propagation. In this study, the single edge notched beam (SENB) method was used to determine the fracture toughness primarily because of its accuracy and simplicity, Caughman et al., (1995); ASTM Designation (1997). The sharp crack requirement is represented by a narrow notch, which is easy to introduce and can be measured accurately Schneider (1991); ASTM Designation (1997).

The fracture toughness results of ormocer-based resin composite (Ceram X) were significantly higher than that of the difunctional methacrylates-based resin composite (Artiste). This may be contributed to the combined effects of nanotechnology with methacrylate modified polysiloxane (ormocers). The presence of the hard nano-ceramic fillers particles (12% wt% , 2-3 nm) distributes the propagating force into many components, causes the crack to curve or dissipate between the particles and becomes energetically unfavourable for crack growth (i.e. hinder further crack growth), Ferracane et al., (1987); Abdel Hamid (2007); Quinn and Quinn (2010). Furthermore, the presence of (Si) particles in the organically modified resin matrix will increase the total inorganic content of the matrix phase. Thus, increasing the fracture toughness values, Hickel et al., (1998); Ceram (2003); Abdel Hamid (2007).

Moreover, the fracture toughness values of the light shade (A1) were significantly higher than that of the dark shade (A3.5) of both resin composites. This may be explained as the curing light can not penetrate along the resin composite thickness of the darker shades, as easily as, the lighter ones due to the presence of different types and content of pigments that control the transmission spectrum of each shade .Therefore, darker shade resin composite had a poor light transmittance into deeper layers, Caughmanet al., (1995); Arikawa et al., (1998); Uhl et al., (2004).

On the other hand, the fracture toughness values of both tested resin composite materials were not significantly different when activated with either LED or halogen light curing units. This could be attributed to the efficient curing and subsequent enhancement of C=C conversion and cross-linking of the polymer matrix of the resin composites cured with the two light curing systems used in this study. As the halogen and the LED produce nearly the same light intensities and wave length ranges which are (700 up to 1000 mW/cm^2 ,430 – 505 nm) and (700 up to 900 mW/cm^2, 430 – 490 nm) respectively, Cesaret al., (2001); Versluiset al., (2004) Therefore, adequate activation of the camphoroquinone (CQ) photo-initiator which is present in both resin composites would occur. In addition, both light curing units produced visible-light with a wave length range that cover the camphoroquinone (CQ) absorbance region of (400 to 500 nm) with a peak at (468 nm). This could explain the good correlation between the absorption spectrum of the camphorquinone and the emission spectrum of the halogen and LED curing units, Cook (1982); Fan et al.,(1987); Caughman et al., (1995); Ferracane and Berge (1995); Deb (1998); Althoff and Hartung (2000); Shivaughnet al., (2009).

Conclusions:

1-Ormocer-based resin composite (Ceram X) exhibited better fracture toughness than those obtained for the di-functional methacrylates-based resin composite (Artiste), irrespective of the light source.

2-Dark shade resin composite exhibited a lowest fracture toughness values regardless to the light source and the resin composite type.

3-The light curing source was not a significant variable for the fracture toughness of resin composites.

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