

An evaluation of the effect of curing systems and water storage on the fracture toughness of two types of composite resin filling materials with different organic matrix composition (in vitro study)

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ABSTRACT

Background: Chipping and bulk fracture are major contributors to clinical failures of composite restorations. Fracture toughness (KIC) quantifies susceptibility for fracture. Halogen light curing units have some specific drawbacks, such as decreasing light output with time. This may result in a low degree of monomer conversion of the composite, which is undesirable, as it can adversely affect mechanical properties including fracture toughness. This study was conducted to evaluate the effect of conventional light cure unit (Astralis – 5), light emitted diode (Radium) and water storage on the fracture toughness of packable composite (Filtek P60) and microfilled hybrid composite (Glacier).

Materials and Methods: KIC was determined by preparing 128 single – edge notched beam test specimens (2X4X20 mm with a 3mm long notch on one edge). The composite specimens were cured by Astralis – 5 or Radium and were subjected to 3 – point bending test (without aging), after 1 day, 7 days, and 28 days storage in distilled water. Student t – test followed by ANOVA (P<0.01) were used to analyze the results.

Results: The fracture toughness values were highly significant when using Radium light cure unit (for both types of composites), and the fracture toughness values for P60 composite were highly significant.

Conclusions: Water aging from 7 days to 28 days didn't produce significant effect in the fracture toughness values for P60 composite, but it led to a highly significant reduction in the fracture toughness values for the Glacier composite. Resin containing Bis – GMA showed higher fracture toughness values than UDMA based resin.

Key words: Fracture toughness, (J Bagh Coll Dentistry 2006; 18(1) 1-5)

INTRODUCTION

Light curing resin based composites have revolutionized clinical dentistry by maximizing working time and minimizing setting time ⁽¹⁾. It's desirable for a dental composite to convert all of its monomer to polymer during the polymerization reaction, since adequate polymerization is a crucial factor in obtaining optimal physical properties and clinical performance of resin composite restorative materials ⁽²⁾.

It's widely accepted that, the mechanical properties of restorative dental composites, including fracture toughness, change with the degree of monomer conversion ⁽³⁾. Mechanical properties such as fracture toughness, may be important properties of restorative materials, that are used where sever biting stress can propagate inherent defects, resulting in inadequate fracture resistance of the materials ⁽⁴⁾. Moreover, failures due to deteriorated mechanical properties and wear may be explained by the influence of moisture from oral environment on the composite, leading to degradation, since the presence of water is of

crucial importance for the deterioration of composite resin materials ⁽⁵⁾. A small change in curing light intensities causes a significant change in degree of conversion within a surface zone (of 2mm deep) of the resin composite, so the presence of poorly polymerized resin layer is undesirable, as it can adversely affect mechanical properties ⁽⁴⁾. To overcome these problems, solid – state light – emitting diode technology has been proposed for curing light – activated dental materials ⁽⁶⁾.

MATERIALS AND METHODS

The materials used in this study are shown in Table 1, and the two types of light curing systems that had been used are shown in Table 2.

One hundred twenty eight single – edge notched beam test specimens were used to determine the fracture toughness (KIC). The test specimen configuration conformed to the American Society for Testing Materials guide lines for the single – edge notched beam specimen ⁽⁷⁾.

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Table 1: Manufacturer's scientific documentation for the composite restorative materials used in this study.

Product	P60	Glacier
Manufacture	3M Dental Products (USA)	Southern Dental Industries (Australia)
Composite Type	Packable	Microfilled hybrid
Resin components	Bis – GMA, UDMA, Bis – EMA	UDMA, DDDMA
Filler Type	Zirconia/ silica	Strontium glass and amorphous silica.
Filler Loading (wt/ vol)	83%wt / 61%vol	77%wt / 62%vol
Curing Time	20s	20s

Table 2: Two types of light curing systems were used.

Traditional Name	Astralis – 5	Radii
Curing Light System	Conventional QTH	Light Emitted Diode
Light Type	Halogen	LED
Curing Tip Diameter	8 mm	7.2 mm
Irradiant Spectrum	400 – 500 nm	440 – 480 nm
Light Intensity	530mW/cm ²	1400mW/cm ²

A custom made stainless steel alloy mold with a sharp blade on one edge was constructed to reproducibly form the composite specimens, into which, the mold consisted of three parts that were assembled and fixed together to form the mold⁽⁸⁾. The dimensions of each specimen used in this study were 2 X 4 X 20 mm with a 3

mm long notch on one edge. Composite resin material was applied in the mold in 2 mm increments, adapted and packed by a plastic instrument⁽⁸⁾ The middle third was activated first, then both of the lateral thirds were activated⁽⁴⁾ (Figure 1).

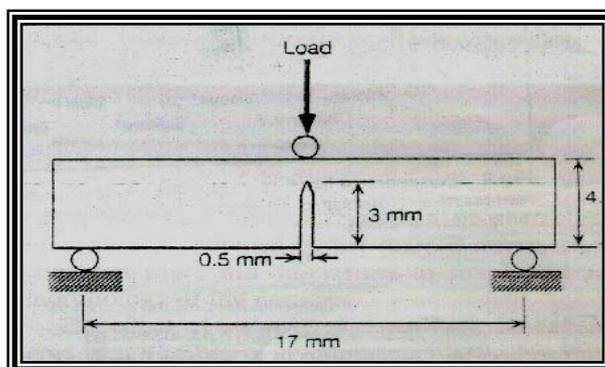


Figure 1: Diagrammatic representation of the single – edge notched beam test specimen

Immediately after each curing of composite resin specimen, the specimen was carefully removed from the mold, and light polymerized on each side for an additional 60seconds⁽⁸⁾. The total 128 composite resin specimens were divided according to the type of composite materials into two groups, each of 64 specimens (packable composite P60 and microfilled – hybrid Glacier composite). Each group was further subdivided into two subgroups of 32 – specimens, according to the type of light curing system used (conventional and light emitted diode), which in turn subdivided into four divisions of 8 – specimens, each according to the aging periods (0–day, 1–day, 7–days, 28–days). The specimen of 0 – day was subjected to the test without aging. A universal testing machine (Instron) was used to apply central load to the specimen in a three – point bending mode at a crosshead speed of 0.5 mm/min. The maximum load at failure was recorded in Newton and calculated in MPa.

Visual examination of the fractured parts was also performed to ensure that the fracture plane was through the notch, and perpendicular to the vertical and horizontal planes through the

center of the specimen⁽⁸⁾. The fracture toughness was calculated with the equation:

$KIC = (P L / b w 1.5) f(a/w)$ where KIC = Stress intensity factor, P = Maximum load at fracture, L = Span (distance between the supports), w = Width of the specimen, a = Crack length, b = Thickness of specimen, f(a/w) = Function of a and w, whose value is obtained from the ASTM standard⁽⁷⁾.

All the specimens of the other subgroups, were immersed in a deionized distilled water inside the sealed polyethylene vials, and maintained inside the incubator at 37±3°C. After each time intervals, the specimens of the other subgroups were removed from water, subjected to three – point bending test as described previously. The data for KIC measurements for the 16 groups were analyzed and compared by Student t – test, and one – way ANOVA (P< 0.01).

RESULTS

Mean and Standard Deviation of fracture toughness values for all groups are presented in Table 3.

Table 3: Mean (M) and standard deviation (SD) of the fracture toughness values for all groups.

Composite	P60		Glacier	
	Astralis-5	Radii	Astralis-5	Radii
Curing unit	Radii		Radii	
Statistics	Mean ± (SD)	Mean ± (SD)	Mean ± (SD)	Mean ± (SD)
0 – day	1.39 ± (0.0307)	1.47 ± (0.0273)	0.88 ± (0.0293)	1.05 ± (0.0346)
1 – day	1.49 ± (0.0245)	1.54 ± (0.0334)	1.19 ± (0.0278)	1.24 ± (0.0245)
7 – days	1.63 ± (0.0200)	1.79 ± (0.0444)	1.37 ± (0.0227)	1.40 ± (0.0363)
28 – days	1.66 ± (0.0293)	1.80 ± (0.0200)	1.18 ± (0.0293)	1.26 ± (0.0273)

Student t–test revealed that, there was a highly significant difference when comparing between the composites cured with Radii or Astralis – 5, and there was a highly significant difference when comparing between P60 composite and Glacier composite (regardless of type of light cure system used). From ANOVA test, it can be seen that, the fracture toughness values increased from 0 – day to 1 – day, and from 1 – day to 7 – days. For P60 composites, there was non significant difference in water aging period from 7 – days to 28 – days, while for the Glacier composite, there was a highly significant reduction in the fracture toughness

values when the specimens were water aged from 7 – days to 28 – days.

DISCUSSION

Fracture toughness is an important characteristic of resin composite restorative materials, because it is related to the resistance of flaw propagation of an existing defect. Such crack propagation can lead to both macroscopic marginal and bulk fracture of resin composite. A fracture toughness parameter, KIC is related to the stress field around a sharp crack. It's a measure of the crack propagation resistance⁽⁹⁾. The results of this study could be explained through the analysis of the total amount of

energy released by each light curing system during polymerization ⁽⁴⁾. To calculate the energy density, the output of the curing source (in mW/ cm²) and the duration of exposure (in seconds) were used. The energy density is expressed in Joules (J) per spot size area (cm²) and is described by the following equation:

$$\text{Energy density} = (\text{Watts} \times \text{seconds}) / \text{cm}^2 = \text{Joules} / \text{cm}^2 = \text{J} / \text{cm}^2 \text{ (4)}$$

When comparing between the two light curing systems, the total energy released from the conventional light curing unit at 20 seconds exposure was 10.60 J/cm² (0.53 W/cm² X 20 seconds), while for the light emitted diode light curing system was 28.00 J/cm² (1.40 W/cm² X 20 seconds), which is higher than the energy released by the conventional light curing system. This could be explained that, the increased light energy for curing composite caused an increase in the conversion of monomer, which leads to enhanced crosslinking density of the polymer, and an increase in the KIC values of the composites ⁽⁴⁾.

For Glacier, the resin matrix is UDMA/DDDMA. The decrease in the fracture toughness could be explained by lower fracture toughness values for the monomer DDDMA, when it was compared with other monomers, ⁽¹⁰⁾. In this study, both types of composites that had been used contain non crystalline filler (glass), and are characterized by high filler loading, which is 61% volume for the P60, and 62% volume for the Glacier, and the fracture toughness values of P60 composite were highly significant than those of Glacier composites, so the filler loading showed no effect on the fracture toughness values in this study.

In this study, KIC increased significantly from 0 – day to 1 – day, and from 1 – day to 7 – days. This could be explained in terms of incomplete polymerization and post cure into which, in photo – activated materials, the polymerization is far from complete when the curing light is turned off, but rather proceeds for 24 hours, to almost 7 days ⁽¹¹⁾, and with high molecular weight monomers such as Bis – GMA or UDMA, there is always an incomplete and significant concentration of unreacted C=C remaining within the resin ⁽²⁾. Such incomplete conversion may leave unreacted monomers that might dissolve in a wet environment ⁽¹²⁾. The increase in physical properties, after water aging, can be attributed to additional cross-linking reactions and increase in the degree of conversion of resin components after light

curing, as the quantity of fillers, which increases physical properties, remains the same ⁽¹³⁾. The increase in the fracture toughness with increase in storage time could be explained that, allowing polymerization process to mature, led to an increase in the fracture toughness values ⁽³⁾. For the P60 composites, there was non significant difference in KIC values from 7 – days to 28 – days. The resin matrix of P60 composites are made up of Bis – GMA /UDMA/ Bis – EMA. These monomers have high monomer weight, and therefore; fewer double bonds per unit area. On the other hand, Bis –EMA is Bis – GMA derived with the hydroxyl groups are removed ⁽¹⁴⁾ and the greater hydrophobicity of the Bis – EMA should reduce the effect of aging ⁽¹³⁾. This could be explained that, the highly cross – linked matrix was simply more resistant to the effect of solvent, even after saturation was attained, and this result in a significant decrease in the solvent permeability of polymer, because they decrease the hole – free volume and the ability of polymer chain for swelling ⁽¹⁵⁾. For the Glacier composite material, the decrease in, KIC from 7 days to 28 days aging in water could be explained that, the water molecules are thought to hydrolyze the bonds at the interfaces, between polymer matrix and inorganic filler particles. This result in interface cracking and hydrostatic stresses, that promote mechanical break down by easier crack propagation (lower KIC) ⁽¹⁶⁾.

REFERENCES

1. Bala O, Uctasli MB, Tiiz MA. Barcoll hardness of different resin – based composites cured by halogen or light emitting diode (LED). *Oper Dent* 2005; 30(1):69 – 74.
2. Yoon TH, Lee YK, Lim BS, Kim CW. Degree of polymerization of resin composites by different light sources. *J Oral Rehabil* 2002; 29: 1165 – 73.
3. Tantbirojn D, Versluis A, Cheng YS, Douglas WH. Fracture toughness and microhardness of a composite: do they correlate? *J Dentistry* 2003; 31:89 – 95.
4. Miyazaki M, Oshida Y, Moore BK, Onose H. Effect of light exposure on fracture toughness and flexural strength of light-cured composites. *Dent Mater* 1996; 12:328-32.
5. Ortengren U, Andersson F, Elgh U, Terselius B, Karlsson S. Influence of pH and storage time on the sorption and solubility behavior of three composite resin materials. *J Dentistry* 2001; 29:35-41.
6. Stahl F, Ashworth SH, Jandt KD, Mills RW. Light – emitting diode (LED) polymerization of dental composites: flexural properties and polymerization potential. *Biomaterials* 2000; 21:1379 – 85.

7. ASTM - Standard E-399-83. Standard test method for plain - strain fracture toughness of metallic materials. In 1984 Annual book of ASTM standards, Philadelphia: ASTM, 1984; p 805 -6.
8. Bonilla ED, Yashar M, Caputo AA. Fracture toughness of nine flowable resin composites. J Prosthet Dent 2003; 89(3):261 - 7.
9. Bonilla ED, Mardirossian G, Caputo AA. Fracture toughness of posterior resin composites. Quintessence Int 2001; 32(3):206 - 10.
10. Indrani DJ, Cook WD, Televantos F, Tyas MJ, Harcourt JK. Fracture toughness of water-aged resin composite restorative materials. Dent Mater 1995; 11:201-7.
11. Hofmann N, Renner J, Hugo B, Klaiber B. Elution of leachable components from resin composites after plasma arc vs. standard or soft - start halogen light irradiation. J Dentistry 2002; 30 : 220 - 2.
12. Yap AUJ, Wee KEC. Effect of cyclic temperature changes on water sorption and solubility of composite restoratives. Oper Dent 2002; 27: 147 - 53.
13. Yap AUJ, Chandra SP, Chung SM, Lim CT. Changes in flexural properties of composite restoratives after aging in water. Oper Dent 2002; 27:468 - 74.
14. Peutzfeldt A. Resin composite in dentistry: the monomer systems. Eur J Oral Sci 1997; 105: 97-116.
15. Ferracane JL, Berge HX .Fracture toughness of experimental dental composite aged in ethanol. J Dent Res 1995; 74(7): 1418 - 23.
16. Pilliar RM, Smith DC, Maric B. Fracture toughness of dental composites determined using the short-rod fracture toughness test. J Dent Res 1986; 65(11):1308-14.