©Operative Dentistry, 2009, **34-6,** 703-708

Effect of Bleaching on Fracture Toughness of Resin Composites

SD Cho • P Bulpakdi
BA Matis • JA Platt

Clinical Relevance

Bleaching after composite placement does not have a significant adverse effect on the fracture toughness of composite materials.

SUMMARY

This study determined the effect of bleaching agents on the fracture toughness of composite materials. Four nanofilled resin composites were evaluated: Filtek Supreme Plus, Tetric EvoCeram, Premise and Esthet-X. Four concentrations of bleaching agents were tested: Opalescence PF 10%, 20%, 35% and 45%. Fifty specimens of each composite conforming to ASTM guidelines for the single edge notch bar-shaped specimen were fabricated in a metal mold. The specimens were stored in artificial saliva at 37°C for a minimum of 24 hours prior to exposing them to the bleaching agents. Ten specimens of each material were used as controls. Forty specimens of each brand were evenly divided into four groups for bleaching agent application (n=10). The resin composites were bleached on both sides for 14 days. The specimens were then subjected to a three-point bending test with a crosshead speed of 0.2 mm per second. The fracture toughness (K_{Ic}) was calculated. The control group K_{Ic} value of Esthet-X was significantly higher than that of the other composites in the controlled groups. Bleaching agents significantly improved the fracture toughness values of Filtek Supreme Plus. The application of bleaching agents did not significantly change the fracture toughness values of the other nanofilled resin composites tested.

*Sopanis D Cho, DDS, MSD, Department of Restorative Dentistry, Indiana University School of Dentistry, Indianapolis, IN, USA

Panida Bulpakdi, DDS, MSD, Department of Restorative Dentistry, Indiana University School of Dentistry, Indianapolis, IN, USA

Bruce Allan Matis, DDS, MSD, professor, Department of Restorative Dentistry, Indiana University School of Dentistry, Indianapolis, IN, USA

Jeffrey A Platt, DDS, MS, professor and director of Dental Materials Division, Department of Restorative Dentistry, Indiana University School of Dentistry, Indianapolis, IN, USA

*Reprint request: 1121 W Michigan St, Indianapolis, IN 46202, USA; e-mail: sdhanvar@iupui.edu

DOI: 10.2341/08-120-L

INTRODUCTION

Restorative dentistry today has evolved from simply repairing the form and function of the human dentition to an increasing demand for lightening the color of teeth. More patients desire a whiter, lighter tooth shade when having restorative treatment done. Advancements in dental materials science have allowed practitioners to perform a variety of procedures to improve the appearance of patients' smiles. Bleaching has gained popularity over the years due to the conservative nature of the treatment and its effectiveness. Several studies confirm the safety and effectiveness of bleaching agents used for teeth whitening.[1]

To improve esthetics, bleaching has been widely used in conjunction with direct restorations and crowns. However, scientific reports of their effects on restorative materials have been limited. The effects of bleaching on the hardness of resin composite in different studies are controversial.[2] One study on microleakage, which used an older dentin bonding system, reported that a bleaching agent had an adverse effect on the marginal seal of the restorations.[3] Studies on color change show that resin composite is somewhat lightened when measured using a colorimeter, but the change was undetectable to the human eye.[4] One study reported that carbamide peroxide had little to no effect on the tensile strength of resin composites.[5]

Fracture toughness is the measure of a material's ability to resist crack propagation. It is considered to be a reliable indicator of the ability of dental materials to resist failure under load.[6] Mode I fracture is the condition wherein a crack opens under normal tensile stress perpendicular to the crack and mode I fracture toughness value is denoted K_{Ic}. Ravindranath and others[7] studied the effects of cyclic loading and environmental aging on the fracture toughness of resin composites. Both aging in a 50/50 alcohol-water mixture and cyclic loading lowered fracture toughness values. The good agreement between the results from the mode I and mixed mode loading conditions from the Ravindranath and others[7] study supported the fact that it was only necessary to characterize the mode I fracture toughness to predict the mixed-mode behavior of the resin composites.

Bonilla and others[8] evaluated the fracture toughness of several resin composites, glass ionomers and amalgam using single-edge notch (SEN) bar-shaped specimens that conformed to ASTM guidelines (Standard E-399). The fracture toughness of titanium-reinforced resin composite (TiCore, Essential Dental Systems, S Hackensack NJ, USA), resin core with fluoride (Fluorocore, Dentsply Caulk, Milford, DE, USA) and spherical amalgam alloy (Tytin, Kerr, Orange, CA, USA) showed comparable fracture toughness values. They concluded that, with the exception of the glass ionomer core materials, all were able to withstand occlusal loads when used as a core material. Another study by the same authors[9] that used the same method found—a packable resin (Surefil, Dentsply Caulk) and a universal resin (Z100, 3M ESPE, St Paul, MN, USA) had the highest fracture toughness values. The other packable resin composites (Glacier, Southern DI; Solitaire, Heraeus Kulzer, South Bend, IN, USA; Bisfil P, BISCO, Schaumburg, IL, USA and Alert, Jeneric/Pentron, Wallingford, CT, USA) were found to range in the mid range with the other universal resin composites (Tetric Ceram, Ivoclar Vivadent, Schaan, Liechtenstein; Bisfil II, BISCO and Prodigy, Kerr). The microfilled resin composite (Heliomolar, Ivoclar) and the microhybrid composite (Charisma F, Heraeus Kulzer) had the lowest fracture toughness values of all the resin composites included. Even though the K_{Ic} values of two packable resins in the current study (Surefil, Dentsply Caulk and Alert, Jeneric/Pentron) were among the highest, the K_{Ic} of the other packable resins was substantially lower than that of the other universal resin composites tested. Regression analysis from the current study revealed a very weak correlation between the K_{Ic} and filler concentration (r^2=0.101). The explanation appeared to be that the filler concentration was not the only factor influencing the K_{Ic} values. Differences between materials with respect to chemical composition of the matrix and filler particles and other characteristics of fillers (size, shape, surface morphology and distribution) were also important factors. Watanabe and others[10] reported the fracture toughness values of six dental composites. They used the Brazilian disk test method and a Zwick testing machine. Modes I and II fracture toughness were reported. The fracture toughness values of the universal hybrid resin (Filtek Z250 and Filtek Supreme, 3M ESPE) were significantly higher than that of the other groups. The intermediate groups consisted of micro-hybrid resin (Point4, Kerr; Venus, Heraeus Kulzer and Gradia Direct, GC). The microfilled resin composite (Durafill) had the lowest mean for the fracture toughness values. Knobloch and others[11] conducted a similar study to compare fracture toughness values of the packable and conventional composites. They used the mini-compact test specimens instead of the single-edge notch (SEN) bar-shaped specimens. In the current study, the packable resin Alert (Jeneric/Pentron) had the highest fracture toughness values. Belleglass (Kerr), SureFil (Dentsply) and Herculite (Kerr) were in the intermediate group, with their fracture toughness values not significantly different from one another. Belleglass is a brand of an indirect ceramic-polymer resin; SureFil is another brand of packable resin, while Herculite is classified as a microhybrid resin. It was interesting that the microfilled resin (Heliomolar, Ivoclar) and the packable resin (Solitaire, Heraeus Kulzer) had the lowest fracture toughness values of all the materials tested and the packable resin (Solitaire) had even significantly lower fracture toughness value than the microfill (Heliomolar). One possible explanation was that both Heliomolar and Solitaire had a similar filler load of 65-66 wt%, which are considerably less than that of other resin composites tested. Other compositions of the resin matrix and fillers in Solitaire were believed to be the factors responsible for the low fracture toughness values. The results from the current study, when considering the wt% of fillers, showed a trend that more heavily filled composites had higher fracture toughness values.

Fleming and Bhamra[12] studied the effects of halogen light variables on flexural strength properties of resin composites. They found that the difference in tip diameter, output intensity and irradiation protocol had no influence on three-point flexural strength and modulus data of the resins tested (Z100, Filtek Z250, Filtek P60 and Filtek Supreme XT, 3M ESPE). The overlapping curing method for specimen fabrication was proven to result in similar mechanical properties in the same resins tested.

The objective of the current study was to determine the effects of bleaching agents on the mode I fracture toughness (K_{Ic}) of nano-particle resin composites commonly used in dental practices. The null hypotheses were:

1) There is no significant difference in the fracture toughness values (K_{Ic}) of resin composites in the control groups.
2) There is no significant difference in the fracture toughness values (K_{Ic}) in the same material between the control and the four bleaching groups.

METHODS AND MATERIALS

Four brands of nanofilled resin composites were evaluated: Filtek Supreme Plus (FSP, 3M ESPE); Tetric EvoCeram (TEC, Ivoclar Vivadent); Premis (PMS, Kerr) and Esthet-X (ESX, Dentsply, York, PA, USA). Shade A2 was used for all resin composites used in the current study. Four concentrations of carbamide peroxide bleaching agents were tested: Opalescence PF 10% (OPF10), 20% (OPF20), 35% (OPF 35) and 45% (OPF45) (Ultradent, South Jordan, UT, USA). The products used, their manufacturers and the lot numbers are listed in Table 1. The materials and their composition are shown in Table 2.

Fifty specimens of each material, conforming to ASTM guidelines for the single-edge notch (SEN) bar-shaped specimen (Standard E-399-83) with dimensions approximately 25 x 5 x 2.5 mm and 2.15 crack length, were fabricated in a metal mold (Figure 1).[13]

In order to facilitate the specimens' removal from the mold, the mold was thoroughly sprayed with a PTFE releasing agent (MS-122 Release agent dry lubricant Miller-Stephenson, Miller-Stephenson Chemical Company, Inc, Danbury, CT, USA) before placing the materials in the mold. The resin composite specimens were cured through a clear Mylar strip in overlapping fashion using the same light-curing unit (Demetron Model VCL 401, Kerr). The light intensity was verified with a radiometer (Model 100 Curing Radiometer P/N 10503, Demetron Research Corporation) to ensure 588 ± 4 mW/cm^2 before each specimen was fabricated. The specimens of each material were kept in artificial saliva at 37°C for 24 hours prior to bleaching. Then, 40 specimens from each group were assigned to four bleaching groups: OPF10, OPF20, OPF35 and OPF45. Plastic trays were fabricated with a bleaching tray material to hold the specimens, while the bleaching materials were being applied (Figure 2).

The bleaching agents were applied on both sides of the 10 specimens for 14 days. The tested specimens were rinsed off daily. The remaining 10 specimens from each composite were stored in artificial saliva at 37°C for 14 days as control groups. All the specimens were subjected to a three-point bending test in an Instron Universal Testing Machine (Instron, Norwood, MA, USA) 24 hours after the two-week period, with a

Table 1: *Composites and Bleaching Agents, Manufacturers, Lot Numbers and Shades*

Materials	Manufacturers	Lot #	Shades
Filtek Supreme Plus (FSP)	3M ESPE	20071030, 20080205 and 20080430	A2B Body
Tetric EvoCeram (TEC)	Ivoclar Vivadent	K25970	A2
Premise (PMS)	Kerr Corporation	2908608	A2
Esthet-X (ESX)	Dentsply	0712261	A2
Opalescence PF 10% (OPF10)	Ultradent Products	#20001.5 051807	
Opalescence PF 20% (OPF20)	Ultradent Products	#20001.5 051807	
Opalescence PF 35% (OPF35)	Ultradent Products	#20001.5 051807	
Opalescence PF 45% (OPF45)	Ultradent Products	B339G	

Table 2: *Materials Used and Their Composition*

Brands	Matrix	Filler
FSP	- BIS-GMA, BIS-EMA(6)	- A non-agglomerated/non-aggregated, 20 nm nanosilica filler - UDMA with small amounts of TEGDMA - Clusters of zirconia/silica particles with size of 5-20 nm fillers. The cluster particle size range is 0.6 to 1.4 μm. The filler loading is 78.5Wt%
TEC	- Dimethacrylates 16.8 Vol%	- Barium glass filler - Ytterbium trifluoride - Mixed oxide - Prepolymers. The filler load is 82.5 Vol%
PMS	- Ethoxylated bis-phenol-A-dimethacrylate - TEGDMA	Trimodal filler system - Prepolymerized filler (PPF), 30-50 μm. - Barium glass, 0.4 μm - Silica filler 0.02 μm The filler load is 84 wt% (71.2 Vol%)
ESX	- BIS-GMA, BIS-EMA - TEGDMA	- Barium boron fluoroalumino silicate glass (<1 μm) more than 70% - Nanofiller silica (0.04 μm) < 3% - Titanium Dioxide less than 1%
OPF10	Carbamide Peroxide 10%, Sodium Fluoride 0.25%, Potassium Nitrate 0.5%	
OPF20	Carbamide Peroxide 20%, Sodium Fluoride 0.25%, Potassium Nitrate 0.5%	
OPF35	Carbamide Peroxide 20%, Hydrogen Peroxide 5.5%, Sodium Fluoride 0.25%, Potassium Nitrate 0.5%	
OPF45	Carbamide Peroxide 20%, Hydrogen Peroxide 9.5%, Sodium Fluoride 1.1%, Potassium Nitrate 3%	

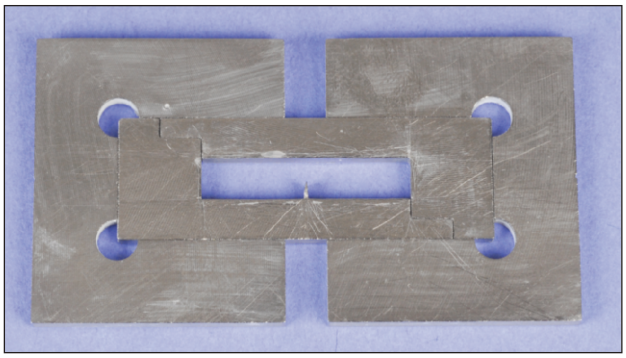

Figure 1*: Metal mold for fabricating specimens.*

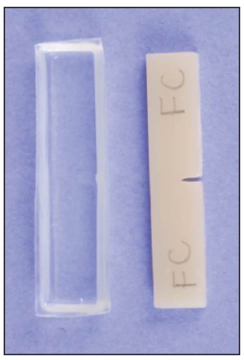

Figure 2*: Plastic trays fabricated with bleaching tray material to hold the specimens during the application of bleaching materials.*

crosshead speed of 0.2 mm/seconds. The loads at fracture were recorded using the Testworks4 computer program (MTS Sintech Renew 1123, Eden Prairie, MN, USA). The fracture toughness values (K_{Ic}) calculated were analyzed statistically using two-way ANOVA and all pairwise multiple comparison procedures (Holm-Sidak).

The results from the current study supported neither of the null hypotheses. The results of the mean fracture toughness values (K_{Ic}) are shown in Table 3. The composite-by-bleaching concentration interaction was significant (p<0.001), therefore, comparisons among the bleaching concentrations are presented by composite, and comparisons among the composites are presented by bleaching concentration. For FSP, the control group had significantly lower fracture toughness values than that of the other concentrations. There were no statistically significant differences between the control group and bleaching concentrations for TEC, PMS or ESX.

RESULTS

Within the control groups, the K_{Ic} values of ESX were significantly higher than that of the other composites

Table 3: *Mean K_{Ic} Values (MPa-m$^{1/2}$) and Associated Standard Deviations in Parenthesis*

Resins	Control	OPF 10	OPF 20	OPF 35	OPF 45
FSP	1.08^{a,A} (0.165)	1.47^{a,B} (0.213)	1.37^{a,B,C,D} (0.179)	1.30^{a,b,C} (0.128)	1.27^{a,D} (0.284)
TEC	1.16^{a,b,A} (0.111)	1.23^{b,A} (0.125)	1.24^{a,A} (0.111)	1.23^{a,A} (0.0829)	1.21^{a,A} (0.111)
PMS	1.25^{b,A} (0.0641)	1.25^{b,A} (0.0612)	1.32^{a,A} (0.0820)	1.22^{a,A} (0.148)	1.25^{a,A} (0.0646)
ESX	1.47^{c,A} (0.0792)	1.41^{a,A} (0.106)	1.36^{a,A} (0.0888)	1.39^{b,A} (0.101)	1.53b,^{A} (0.0913)

Individual group means signified by the same lower case letters in each column or by the capital letters in each row are not significantly different for pairwise comparison using the Holm-Sidak method at the overall significance level = 0.05.

tested. The K_{Ic} values of FSP were significantly lower than that of PMS but not TEC.

For the 10% bleaching concentration, FSP and ESX had significantly higher fracture toughness than PMS and TEC. There was no statistical difference between the K_{Ic} values of FSP and ESX. The K_{Ic} values of PMS and TEC were not significantly different.

For the 20% bleaching concentration, there were no significant differences among the composites.

For the 35% bleaching concentration, ESX had significantly higher fracture toughness values than PMS and TEC. There were no other significant differences among the other composites tested.

For the 45% bleaching concentration, ESX had significantly higher fracture toughness values than that of the other composites tested. There were no other significant differences among the other composites.

DISCUSSION

Dental resin composites consist of a polymerizable resin matrix, reinforcing glass particle fillers and silane coupling agents. The polymerizable resin matrix generally contains one or more monomers. Examples of resin matrix are bis-phenol-A-diglycidyl dimethacrylate (Bis-GMA), urethane dimethacrylate (UDMA) and triethylene glycol dimethacrylate (TEGDMA). Various inorganic materials, such as glass fillers, are added as reinforced components.[14] Even though all the resin composites included in the current study utilized nanofiller technology, there were significant differences in the fracture toughness values (K_{Ic}). This result supported data from the past that the materials categorized in the same group as nano-particle resin composites do not always have similar physical and mechanical properties. Previous studies reported various three-point flexural strength data within the same brands of resin composite.[15-16] In the control groups, ESX had significantly higher fracture toughness values than the others and PMS had significantly higher fracture toughness values than FSP. There were no significant differences between the other groups. The differences in fracture toughness values are likely the result of various compositions that exist between the individual brands as shown in Table 2.

The results of the current study showed a significant increase in fracture toughness values in the FSP groups after bleaching but not in the other materials tested. The most significant improvement in fracture toughness values was seen when bleached with 10%, followed by 20%, 35% and 45%, respectively.

It is interesting to see that FPS had the lowest K_{Ic} values in all the control groups but had significantly improved strength after bleaching. When comparing the K_{Ic} of resin composites after bleaching with 10%, FSP was significantly stronger than both TEC and PMS. To eliminate the possibility of misleading results from an isolated batch, FSP was randomly used from three batches as shown in Table 1. The low values of fracture toughness of FSP in a control group may suggest incomplete polymerization. This result is consistent with the peroxides of the bleaching agent acting as an additional initiator of the polymerization of the matrix. Increased initiation would be expected to increase the average molecular weight of the resin matrix, enhancing its mechanical properties. Initial maximal polymerization of the control groups in other composites resulted in no change of fracture toughness values after bleaching.

The leaching of fillers from resin composites has been recognized as occurring within aqueous environments.[17-18] It could be hypothesized that increasing the concentration of the peroxide is associated with a degradation process that would result in decreasing fracture toughness. A simple linear regression analysis on the means of the bleached groups for FSP indicates an R2 value of 0.944. This would indicate a potentially undesirable degradation of the material with prolonged exposure to high concentration bleaching agents. Further investigation is required in order to understand the observations.

CONCLUSIONS

Of the nanofilled resin composites included in the current study, Esthet-X had a significantly higher fracture toughness value than the others. Bleaching had a significant effect in increasing the fracture toughness value on Filtek Supreme Plus but not on the other composites. The practice of bleaching after placement of the composite restoration does not compromise the fracture toughness property of the resin composites tested.

Acknowledgements

The current study was funded, in part, by the Professional Development Fund, Indiana University School of Dentistry, and Ultradent Products, Inc. The authors thank Ultradent Products, Inc for providing the bleaching agents and Dentsply for providing Esthet-X, which was used in this study.

(Received 10 December 2008)

References

1. Haywood VB (1992) History, safety, and effectiveness of current bleaching techniques and applications of the nightguard vital bleaching technique *Quintessence International* **23(7)** 471-488.
2. Yap AUJ & Wattanapayungkul P (2002) Effects of in-office tooth whiteners on hardness of tooth-colored restoratives *Operative Dentistry* **27(2)** 137-141.
3. Crim GA (1992) Post-operative bleaching: Effect on microleakage *American Journal of Dentistry* **5(2)** 109-112.

4. Swift EJ & Perdigão J (1998) Effects of bleaching on teeth and restorations *Compendium of Continuing Education in Dentistry* **19(8)** 815-820.
5. Cullen DR, Nelson JA & Sandrik JL (1993) Peroxide bleaches: Effect on tensile strength of composite resins *The Journal of Prosthetic Dentistry* **69(3)** 247-249.
6. Kovarik RE & Muncy MV (1995) Fracture toughness of resin-modified glass ionomers *American Journal of Dentistry* **8(3)** 145-148.
7. Ravindranath V, Gosz M, De Santiago E, Drummond JL & Mostovoy S (2007) Effect of cyclic loading and environmental aging on the fracture toughness of dental resin composites *Journal of Biomedical Materials Research Part B: Applied Biomaterials* **80(1)** 226-235.
8. Bonilla ED, Mardirossian G & Caputo AA (2000) Fracture toughness of various core build-up materials *Journal of Prosthodontics* **9(1)** 14-18.
9. Bonilla ED, Mardirossian G & Caputo AA (2001) Fracture toughness of posterior resin composites *Quintessence International* **32(3)** 206-210.
10. Watanabe H, Khera SC, Vargas MA & Qian F (2008) Fracture toughness comparison of six resin composites *Dental Materials* **24(3)** 418-425.
11. Knobloch LA, Kerby RE, Seghi R, Berlin JS & Clelland N (2002) Fracture toughness of packable and conventional composite materials *The Journal of Prosthetic Dentistry* **88(3)** 307-313.
12. Fleming GJP & Bhamra GS (2008) Effects of halogen light irradiation variables (tip diameter, irradiance, irradiation protocol) on flexural strength properties of resin-based composites *Journal of Dentistry* **36(8)** 643-650.
13. American Society for Testing of Materials (ASTM) (1983) Standard test method for plain-strain fracture toughness of metallic materials E399 *1983 ASTM Book of Standards* 518-551.
14. Drummond JL (2008) Degradation, fatigue, and failure of resin dental composite materials *Journal of Dental Research* **87(8)** 710-719.
15. Beun S, Glorieux T, Devaux J, Vreven J & LeLoup G (2007) Characterization of nanofilled compared to universal and microfilled composites *Dental Materials* **23(1)** 51-59.
16. Mitra SB, Wu D & Holmes BN (2003) An application of nanotechnology in advance dental materials *Journal of the American Dental Association* **134(10)** 1382-1390.
17. Söderholm KJ, Zigan M, Fischlschweiger W & Bergman M (1984) Hydrolytic degradation of dental composites *Journal of Dental Research* **63(10)** 1248-1254.
18. Söderholm KJ, Mukherjee R & Longmate J (1996) Filler leachability of composites stored in distilled water or artificial saliva *Journal of Dental Research* **75(9)** 1692-1699.