Fracture toughness of seven resin composites evaluated by three methods of mode I fracture toughness ($K_Ic$)

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This study compared the fracture toughness values of seven resin composites and analyzed the consistency of data values using three methods: compact tension, three point and four point bending for Mode I failure. Seven resin composites were selected: Estellite Sigma Quick, Esthet X HD, Filtek Supreme XTE, Heliomolar, Ice, Rok, and Vit-l-escence. For each material, 18 specimens ($n=6$ for each test) were prepared. Maximum load to failure was recorded using a universal testing machine and fracture toughness was calculated. There was a direct significant correlation among all tested methods. Rok showed the highest and Estelite the lowest $K_Ic$ values. SEM of the fractured surface of compact tension method showed propagation of the cracks from stresses concentrating at the corner of the notch and the surface of the sample. Four-point test gave the largest range in $K_Ic$ and was a simple method to discriminate between the resin composite values of $K_Ic$.

Keywords: Fracture toughness, Four-point method, Three-point method, Compact tension method, Resin composite

INTRODUCTION

Resin composites are now commonly used for all types of restoration; however, they tend to be brittle and this shortcoming relates to a sensitivity to flaws and defects that increases susceptibility to catastrophic failure3. New formulations of resin composites are continually appearing on the market with different mechanical properties.

The ability of a restoration to tolerate fracture partly lies in material composition and filler volume. A higher filler loading has been shown to result in higher fracture toughness up to a threshold value of $\sim 55$ to $57\%$ filler loading2,4. Kim et al. in their study of the effect of filler loading and morphology on the mechanical properties of resin composites (filler loading rate of 41.24 to 59.05 vol%), determined the maximum toughness at the threshold level of approximately $55\%$ filler. Ilie et al. in their study of fracture toughness of restorative materials also reported an increase in $K_Ic$ with the volume fraction of fillers until a critical volume fraction of $57\%$.

The physical and mechanical properties of resin composite critically affect the performance in the oral cavity. Due to the wide use of resin composites, they should reproduce the strength and wear limits similar to natural tooth structure being able to resist occlusal forces including tensile, compressive, and shear stresses. One of the common mechanical tests used to assess brittle restorative materials is fracture toughness.

$K_Ic$ is an important measure of a material’s properties as it indicates the largest amount of stress that a material can bear prior to failure. A variety of fracture toughness testing methods have been used to evaluate the relative fracture toughness of resin composites5. These tests include four-point bending6,7, single-edge-notched beam8, chevron notch, compact tension9,10, and the indentation hardness method11, all resulting in a range of values of $K_Ic$, even for the same material11,9. These methods require different specimen geometries related to the size and shape of the specimens, size of the crack, and loading configuration.

Due to the complexity of the forces that direct restorations are subjected to in the oral cavity, it is not easy to select a suitable method for testing fracture toughness of resin composites. Several studies attempted to establish some clinically reliable testing protocols for evaluating the fracture toughness of brittle dental materials12-15. One of the most reported reliable methods is the single edge V-notched beam (SEVNB) which has been used in the ceramics standard, ISO 687216. It has been shown that SEVNB method is a reproducible approach17. However, the reported values show a large discrepancy in the fracture toughness values obtained from this method18; this could be related to the difficulties in preparing the V-notch that may vary in size and consequently lead to different $K_Ic$ values.

This study aimed to compare fracture toughness of seven resin composites using three different methodologies to see if outcomes were similarly ranked and of the same order of magnitude. The fractographic features of the surface were evaluated using scanning electron microscopy (SEM). The null hypothesis is that fracture toughness values are not similarly ranked using three different methodologies and that the geometry of
the testing method (Mode I) has no effect on the fracture toughness value.

MATERIALS AND METHODS

Specimen preparation
1. Four-point test method

Seven resin composites of shade A2 were selected (Table 1) with the particle filler content ranging from 66.7 to 82.3 wt% (46 to 67.7 vol%). A custom-made, brass and aluminium mould with a notch centrally placed was used to prepare the specimens (Fig. 1-A). Six bar-shaped specimens of 25 mm long×2 mm thick and 5 mm width (Fig. 1-B) were prepared from each material by filling the mould and pressing between transparent plastic strips and glass plates to extrude the excess material. The materials were light cured through transparent strips according to the manufacturers’ instructions for 40 s (three exposures over equal areas) using a light-polymerizing LED unit with a wavelength range of 440–480 nm at an output of 1,500 mw/cm² (Radii plus LED, SDI, Bayswater, Vic, Australia). Each specimen was

Table 1 Materials description

| Materials     | Manufacturer                  | Type               | Resin                   | Filler’s type, size and %          | Lot #  |
|---------------|-------------------------------|--------------------|-------------------------|-----------------------------------|--------|
| Estelite Sigma Quick | Tokuyama Dental, Tokyo, Japan | Submicron filled composite | Bis-GMA, TEGDMA | SiO₂, ZrO₂ (200 nm), PFSC (average 0.2 μm); 78 wt% (63 vol% ) | E033   |
| Esthet X HD   | Dentsply, Caulk, USA          | Microhybrid Composites | BIS-GMA, BIS-EMA, TEGDMA | Barium, FSG (<1 μm), NFS (0.04 μm), TD (nanofiller); 60 vol% (76 wt%) (0.2 to 2.5 μm) | 1111041|
| Filtek Supreme XT | 3M ESPE St. Paul, MN, USA    | Nanohybrid Composites | Bis-GMA, UDMA, TEGDMA, Bis-EMA | 63.3 vol% (78.5 wt%) SF, ZF, AZSCF (5 to 75 nm) | N395233|
| Heliomolar    | Ivoclar Vivadent, Schaan, Liechtenstein | Microfilled Composite | BisGMA, UDMA, D DDDMA | 0.04–0.2 μm, 46 vol% (66.7 wt%) | N04438|
| Ice           | Southern Dental Industries, Vic, Australia | Nanohybrid Composite | UDMA/BisEMA/TEGDMA | SAS, AS (0.04–1.5 μm), Average1.0 μm, 61 vol% (78 wt%) | 12096SN|
| Rok           | SDI, Vic, Australia           | Hybrid Composite    | UDMA, TEGDMA, Bis-EMA | SAS, AS, (0.04–2.5 μm); 67.7 vol% (82.3 wt%) | 120844|
| Vit-l-escence | Ultradent Products, USA       | Microhybrid Composite | Bis-GMA | Bis-GMA, Average particle size (0.7 μm); 58 vol% (75 wt%) | B56VM  |

SF=Silica filler, ZF=Zirconia filler, AZSCF=Aggregated zirconia/silica cluster filler
SAS=Strontium alumino silicate, AS=amorphous silica, FSG=fluoroalumino silicate glass, NFS=Nanofiller silica, TD=Titanium Dioxide, PFSC=Prepolymerized filler of silicacomposite, DDDMA=Decandioldimethacrylate.

Fig. 1 A: Custom-made, brass and aluminium mould with a centrally placed notch, B: Bar-shaped specimen.
removed from the mould and light-cured on the opposite side for an additional 3×40 s. Before the measurement of \( K_c \), the specimens of each material were stored in distilled water at 37ºC for 24 h.

After polymerization, in order to obtain a flat surface, the edges of the specimens were ground by gentle wet grinding using 1000-grit silicon carbide paper. A new razor blade was used under hand pressure to create a sharp crack in the notch. Crack length was measured and recorded for each specimen using a stereomicroscope (LV 150 Eclipse, Nikon, Japan) at 60× magnification. The width and height of each specimen was measured using a digital calliper with accuracy of ±0.1 mm. Figure 2-A displays the specimen geometry for determination of fracture toughness by the four-point bend method. The specimens were placed in the universal testing machine (Zwick/Roll Z020, Zwick, Germany) using a four-point bend test jig, loaded at a crosshead speed of 0.5 mm/min, and calibrated using the internal calibration. The maximum load at specimen failure was recorded and the \( K_c \) (MPam\(^{0.5}\)) was calculated using the following equation:

\[
K_c = \frac{F \cdot S_{1} - S_{2}}{B \cdot w^{1.5}} \cdot Y \]

Where:
- \( F \) = fracture load,
- \( S_{x} \) = span (\( x = 1 \): outer span; \( x = 2 \): inner span),
- \( B \) = specimen width,
- \( a/w \) = notch depth,
- \( w \) = specimen height,
- \( Y \) = stress intensity shape factor

2. Three-Point test [Single-edge notched beam method (SENB)]

The same mould and method as the four-point test was used to prepare six specimens for each of the seven materials (Fig. 2-B). The specimens were placed in the universal testing machine (Zwick/Roll Z020, Zwick) using a three-point bend test jig, loaded at a crosshead speed of 0.5 mm/min, and calibrated using the internal calibration. The maximum load at specimen failure was recorded and the \( K_c \) (MPam\(^{0.5}\)) was calculated using the following equation:

\[
K_c = \frac{F}{B} \cdot \frac{S_{c}}{w^{3/2}} \cdot f(c/w)
\]

\( f(c/w) = 2.9(c/w)^{1/2} - 4.6(c/w)^{3/2} + 21.8(c/w)^{5/2} - 37.6(c/w)^{7/2} \)

\( F = \text{Maximum Load}, \ B = \text{Specimen width}, \ S = \text{Supporting span}, \ w = \text{Specimen height}, \ c = \text{notch length}, \ f(c/w) = \text{a function of } c \text{ and } w \)

3. Compact Tension (CT) test method

A custom-made, polytetrafluorethylene (PTFE) split mould was used to prepare six disk-shaped specimens for each of the seven materials. A schematic of the specimen and details of the dimensions were shown and described in previous studies\(^9,17\). A pre-crack was created in the mini-compact specimen with a razor blade, as described by Kovarik and Fairhurst\(^18\) in accordance with ASTM Designation: E399-83\(^19\). The split mould was assembled.
using guide screws. The mould was filled with the resin composite and covered by plastic matrix strips and glass plates under gentle hand pressure in order to extrude the excess material. A razor blade was used to create a sharp pre-crack in the notch during polymerization of the resin composite. The materials were cured according to the manufacturers’ recommended exposure times, using the same LED curing light as explained above.

The specimens of each material were stored in distilled water at 37°C for 24 h and then ground by gentle wet grinding using 1000-grit silicon carbide papers in order to adjust the thickness to 2 mm ±0.2 and measured using a digital calliper with accuracy of ±0.1 mm. Faulty specimens having voids or cracks were discarded.

The specimens were secured in universal testing machine (Zwick/Roll Z020, Zwick) using guide pins placed through the specimen’s holes (Fig. 3-C). Tensile loading was applied at a crosshead speed of 0.5 mm/min; the maximum load at failure was recorded and the $K_{\text{IC}}$ (MPa.m$^{0.5}$) was calculated using the following formula:

$$K_{\text{IC}} = \frac{F}{BW^{0.5}} \cdot f\left(\frac{a}{w}\right)$$

Where:

$$f\left(\frac{a}{w}\right) = \frac{(2+\frac{a}{w})[0.76+4.8\frac{a}{w}-11.58(\frac{a}{w})^2+11.43(\frac{a}{w})^3-4.08(\frac{a}{w})^4]}{(1-\frac{a}{w})^{1.5}}$$

$F$=maximum load at specimen fracture, $f(a/w)$=function of $a$ and $w$
$B$=specimen thickness, $W$=dimension from the un-notched edge of the specimen to the plane centreline of the loading holes.

**Scanning electron microscopy**

To observe the fractured surface in each testing method, two randomly selected specimens of each group were examined under an environmental scanning electron microscope (ESEM, FEI Quanta, OR, USA) with original magnification range of 100 to 2,000 times (Figs. 3a–c). Samples were sectioned parallel to the fractured surface using a diamond peripheral saw (Minimat 230CS, Struers, Copenhagen, Denmark) and stuck to a SEM stub with carbon conductive tape.

**Data analysis**

The collected data were analyzed using the SPSS package (version 18, SPSS, Chicago, IL, USA). The normality assumption was assessed using Kolomogorov-Smirnov test. The Pearson correlation coefficient was performed to evaluate the possible correlation between the three tests. Then, one-way ANOVA with post hoc Tukey’s test was used for each method to compare the fracture toughness of the seven tested materials.

**RESULTS**

The mean $K_{\text{IC}}$ values and standard deviations from all tests for the seven resin composites are presented in Table 2. The values obtained from CT test were lower than those obtained by four-point or three-point loading tests. Figures 4–6 shows force-displacement curve for representative specimens of the each material of CT, three point and four point tests respectively.

**Comparison among the three methods**

The Pearson correlation coefficient analysis showed a strong direct significant correlation between the three methods; between CT and three-point test ($p<0.004$, $r=0.509$); CT and four-point ($p<0.001$, $r=0.438$) and between four-point and three-point ($p<0.001$, $r=0.614$). The lowest values were obtained when using the compact tension method.

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Fig. 3  
(a) Compact tension fracture test of Esthet X shows “artificially introduced crack interface” (A), mirror region (B), mist region (C), hackle region (D) and voids (E),  
(b) Three-point fracture of Filtek Supreme showing “artificially introduced line notch interface” (A) and voids (B),  
(c) Four-point fracture of Heliomolar shows “artificially introduced line notch interface” (A) and voids along an internal line defect (B).
Table 2 Means $K_I$ (MPa.m$^{0.5}$) and SD(±) of all materials in 3 testing methods $n=6$

| Materials          | Compact tension | Four-point bending | Three-point bending |
|--------------------|-----------------|--------------------|---------------------|
| Estellite Sigma Quick | 0.51 (0.15)$^a$ | 0.90 (0.17)$^a$   | 0.91 (0.17)$^p$    |
| Esthet. X HD       | 0.56 (0.07)$^a$ | 1.45 (0.24)$^{bc}$| 1.34 (0.20)$^{bc}$ |
| Filtek Supreme XTE | 0.58 (0.05)$^a$ | 1.48 (0.20)$^{bc}$| 1.45 (0.13)$^{bc}$ |
| Heliomolar         | 0.49 (0.06)$^a$ | 1.05 (0.19)$^{cd}$| 1.13 (0.08)$^{cd}$ |
| Ice                | 0.56 (0.06)$^a$ | 1.28 (0.24)$^{cd}$| 1.16 (0.17)$^{cd}$ |
| Rok                | 0.75 (0.08)$^b$ | 1.75 (0.09)$^r$   | 1.48 (0.07)$^r$    |
| Vit-l-escence      | 0.53 (0.10)$^a$ | 1.17 (0.07)$^{bc}$| 1.08 (0.15)$^{bc}$ |

Different lower case letters indicate that the values are statistically significant for each column ($p<0.05$).

Fig. 4 Force-Displacement curve for representative specimens of the each material of CT test.

Fig. 5 Force-Displacement curve for representative specimens of the each material of three-point test.

Fig. 6 Force-Displacement curve for representative specimens of the each material of four-point test.

Comparisons of fracture toughness among different materials

One-way ANOVA and post hoc Tukey’s test showed a significant variation among materials for the different tests. For CT test, the differences among materials were not significant except for Rok (0.75 MPa.m$^{0.5}$) which showed a significantly higher value than all other materials. In four-point test, Rok (1.75 MPa.m$^{0.5}$), Filtek Supreme (1.48 MPa.m$^{0.5}$) and Esthet X (1.45 MPa.m$^{0.5}$) demonstrated the highest values, whilst Estellite Sigma Quick (0.90 MPa.m$^{0.5}$) and Heliomolar (1.05 MPa.m$^{0.5}$) the lowest. For three-point test, Rok, Filtek Supreme and Esthet X showed the highest values, respectively, in contrast with Estellite Sigma Quick, and Vit-l-escence which showed the lowest values.

Results of SEM examination

The SEM fractographic examination is presented in Figs. 3a–c. Figure 3-a illustrates an example of the fractured surface of CT method (in Esthet X) indicating...
flaws and characteristic crack initiation point where a mirror, mist and hackle region at the corner of the compact tension with the notch and the disk surface can be seen. Figure 3-b shows an image of the three-point fracture test of Filtek Supreme showing a line of voids just below the introduced notch (artificial crack) interface on the fractured surface. Figure 3-c shows the four-point fracture test displaying internal defects in Heliomolar and as a line defect associated with voids crossing the field of view that may account for the low fracture toughness values for this material.

**DISCUSSION**

The Mode I fracture toughness ($K_c$) is the lowest stress at which catastrophic crack propagation will occur due to its tensile opening of the crack. $K_c$ is an important material property as it represents the ability of a material to resist crack propagation from an existing flaw.

In this laboratory study, three methods of determining fracture toughness values for Mode I (tensile force) were evaluated for different types of resin composites. The results showed a strong direct significant correlation between all tested methods with the lowest values for CT method. The low value found for the CT fracture test compared to the three- and four-point tests could be due to the concentration of tensile and possibly torsional or shear stresses occurring at the edges (corners) of the line notch interface with the surface of the disk. Corner stresses developed at flaws present at the introduced crack interface of the CT samples (Fig. 3-a).

The tensile stress due to the mirror size (Fig. 3-a) can be calculated from the formula: $\sigma = \frac{\sqrt{aR}}{A}$. Where $A$ is a constant of the material; $R$ is the mist hackle radius and $\sigma$ is the stress. "A" is equal to $\sim 2.6$ MPA $\sqrt{m}$ according to Quinn for 85 wt% of filler in a Bis-GMA/TEGDMA composite. This implies that the localized tensile stress is concentrated at the notch/surface corner and not along the length of the interface. The stress value to initiate the crack was approximately 106 MPa. This is consistent with the flexural strength reported for Esthet X.

It appears that it is difficult to obtain a uniform stress along the whole line of the introduced notch and that stresses will preferentially concentrate at the corners of the disk. Consequently, this produces low fracture toughness values for this rigid CT geometry compared to the three and four-point fracture methods.

Dental composites are multipurpose materials which have grown fast since the materials were first introduced to the market. Modification of the filler types, reduction of the filler particle size but also an associated increase in size range that has allowed an increase in the filler loading are the most significant changes that have occurred since their introduction in the 1960's. In resin composite with particles ranging from 0.04 to 0.20 μm, classified as micro-filled, about 50% of the volume of the material is resin which provides excellent surface smoothness. However, their physical and mechanical properties are inferior to those of hybrid resin composites as observed in our study for Heliomolar with its low filler content.

More recently, the description “nano-hybrid” has been marketed. The distinction between micro-hybrids and nano-hybrids is not always clear, perhaps due in part to the way they are marketed. After all, even micro-hybrids contain a small fraction of nano-sized (sub 100 nanometer) particles. Manufacturers add nanoparticles to micro-hybrids to fill the resin filled gaps between the larger particles. This can result in improved esthetic quality. There is, however, a limit to the amount of nanoparticles that can be added before the handling becomes too stiff and therefore unworkable clinically.

In our study as shown in Table 2, the hybrid resin composite (Rok) with the highest filler vol% 67.7 (82.3 wt%) and the largest filler sizes (2.5 μm) showed significantly higher values in all tests compared to the nano-hybrid, micro-hybrid and microfill composites. On the other hand, when the materials were tested by compact tension method, Heliomolar with the lowest filler content of 46 vol% (66.7 wt%) showed the lowest $K_c$ value (0.49 MPam$^{0.5}$) followed by Vit-l-escence (0.53 MPam$^{0.5}$) with filler content of 58 vol% (75 wt%). Apart from Estelite, all other materials showed a direct positive relationship between $K_c$ values and filler vol% (wt%), i.e. (within the range investigated) as the filler content increased, the fracture toughness also increased. However, the four point loading method showed the greatest change in fracture toughness as a function of filler loading.

The wide distribution of particle sizes leads to a higher filler loading and therefore resultant higher fracture toughness. Improvements in resin composites have been directed at increasing the filler content in the resin matrix and reducing the filler particle size range. There is a correlation between the filler particle size and the maximum filler volume that can be incorporated in the resin matrix. Similarly, the particle morphology is believed to have an effect on the fracture toughness values so that the percentage of the filler volume is influenced by filler morphology. There was no significant difference between nano-hybrid and micro-hybrid which contain a mixture of larger particles and smaller sub-micrometre sized particles, usually amorphous or colloidal silica. Particle size on average is typically below 3 micrometre but above 0.04 micrometres.

**CONCLUSION**

Within the limitations of this study, the four-point and three-point tests gave similar $K_c$ values. Lower values were observed for the compact tension method which may be due to corner stresses generated during testing. All the methods of testing showed similar ranking order between the resin composites tested. Of the systems tested the four-point method provides a more discriminating method to determine the fracture toughness ($K_c$) value for resin composites with different percent filler content.
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