

The Role of Home Bleaching Agent on the Fracture Toughness of Resin Composites Using Four-Point Bending Test

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Abstract

Statement of Problem: Resin composites are a common type of tooth coloured restorative materials. These materials are brittle and their major shortcomings are sensitivity to flaws and defects, low tensile strength, and susceptibility to catastrophic failure. The role of home bleaching agents on the fracture toughness of resin composites using four-point bending test is scanty.

Objectives: To compare the fracture toughness (K_{Ic}) of resin composites on a four-point bending test and to assess the effect of distilled water and a home bleaching agent on the resistance of the materials to fracture.

Materials and Methods: seventy-two bar-shaped specimens were prepared from three materials: Rok (SDI), Estelite (Tokuyama), and Vit-l-escence (Ultradent) and divided into three groups. Two groups were assigned as "control" and conditioned in distilled water at 37°C for 24 hours or 21 days, respectively. The specimens in the third group (treatment) were stored in distilled water for 21 days and bleached using Polanight (SDI) for 2 hours daily. For each material, a total of 24 disc-shaped specimens were prepared and after each time interval loaded in a four-point bending test using a universal testing machine with a crosshead speed of 0.5 mm/m. The maximum load to specimen failure was recorded and the K_{Ic} was calculated.

Results: Statistical analysis using two-way ANOVA showed a significant relationship between materials and treatment ($P < 0.05$). Tukey's test showed that after 24 hours of immersion in distilled water, K_{Ic} was not significantly different between materials; Rok revealed the highest value followed by Estelite and Vit-l-escence. The bleaching agent significantly decreased the K_{Ic} values of Estelite and Rok while it did not affect that of Vita-l-escence. Immersion in distilled water for all resin composites caused a significant decrease in K_{Ic} .

Conclusion: The fracture toughness of the resin composites was affected by the bleaching agent and 21day immersion in distilled water.

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Introduction

Due to high demands for a better smile and aesthetics, resin composites are currently used for the restoration

of virtually all forms of cavity preparation. It is difficult for clinicians to choose a resin composite, which achieves excellent physical, chemical and mechanical properties as well as high polishability [1]. Composites

with high filler levels have better physical, chemical and mechanical properties, but clinically, composites with small filler particles are easier to finish [1]. Hybrid and nano-hybrid composites are normally used for posterior restorations where high mechanical strength and fracture resistance are important [1,2].

Haywood and Heymann introduced dental home bleaching using carbamide peroxide in 1989 [3]. This treatment has become a popular conservative option for patients seeking improved aesthetics in a relatively short period of time at low cost. Home bleaching is prescribed by a dentist, but is performed by the patient at home using custom-made plastic trays that hold the bleaching agent against the patient's teeth for a specified time according to the manufacturers' instructions. It has been reported that bleaching lasts approximately six months to two years; the teeth then often return to their original colour [4]. As previously reported by Swift et al. [5] 83.3% of patients had a shade change of two or more units, and the result remained statistically significant at 2 years.

All composite restorations are subjected to three processes that affect their longevity; moisture, shrinkage during polymerization and wear. These processes can lead to water sorption and deterioration of the silane coupling agent [6]. Water sorption by a resin composite depends on the matrix resin, the filler and the properties of the interface between the matrix and the filler [7]. Water sorption by polymers is a diffusion process and occurs mostly into the resin matrix [8, 9]. The greater the resin content, the more water is absorbed [8,10-12]. Excessive water uptake can promote breakdown causing filler-matrix debonding [12]. Water exposure may decrease the life of resin composites by silane hydrolysis and microcrack formation [12]. Hence, the outcome of water sorption may alter the strength and fracture toughness of resin-based materials. It has been reported that aging in water leads to a significant decrease in the fracture toughness of resin composites in the first 6 months with minimal changes from 6-24 months [13,14]. To determine the durability and strength of restorative materials, fracture toughness

(K_{Ic}) has been a key focus.

Determination of K_{Ic} is technically sensitive, and the values obtained and subsequent rankings may differ depending on the techniques and the procedures used [15]. Different test methods have been established to evaluate the K_{Ic} values of restorative materials. It has been shown that fracture toughness is dependent upon several variables including crack-tip sharpness and specimen geometry [15]. It is therefore concluded that although the double torsion test (4-point bending) is the most sensitive technique, and also the highest material consuming, it provides the most information about crack initiation and propagation and may be indicative of the 'true' fracture toughness of restorative materials [15,16].

However, limited information is available on the effect of bleaching agents on the fracture toughness of resin composites using the 4-point bending test [17]. The objective of the present study was to place various commercially available resin composites into distilled water at 37°C and determine (1) the effect of immersion time in distilled water, with and without exposure to 10% carbamide peroxide using a double torsion test; (2) if a relationship exists between the mechanical properties and the material composition of the material tested. The null hypothesis is that bleaching treatment has no effect on fracture toughness of resin composites, also that the storage media and time do not affect mechanical properties; in other words, there is no relationship between the mechanical properties and material composition.

Materials and Methods

Three resin composites of shade A2 and one home bleaching agent were selected (Table 1).

A custom-made, brass and aluminium mould with a centrally placed notch was engaged to prepare specimens (Figure 1).

Figure 2 shows a schematic representation of the specimen configuration along with all dimensions in accordance with ASTM Designation: E399-83 [18].

Seventy two bar-shaped specimens were made

Table 1: Materials with their technical information

Materials	Manufacturer	Type	Resin	Filler's type and %	Lot #
Rok	SDI, Vic, Australia	Hybrid composite	UDMA, TEGDMA, Bis-EMA	SAS, AS, (0.04 -2.5 μ m); 82.3 Wt% (67.7 Vol %)	120844
Vit-I-escence	Ultradent Products, Inc, USA	Microhybrid composite	Bis-GMA	Average particle size (0.7 μ m); 75 Wt% (52 Vol %).	B53PL
Estelite Sigma Quick	Tokuyama, Dental Corporatio, Tokyo	Submicron filled composite	Bis-GMA, TEGDMA	SiO ₂ , ZrO ₂ (200 nm), PFSC (average 0.2 μ m); 78Wt% (63 Vol%)	082E61
Polanight	SDI, Victoria, Australia	Home Bleaching	10-22 Wt% Carbamide Peroxide, <40Wt% additives, 30Wt% glycerol, 20Wt% water, 0.1% Flavour		P120309

SAS= Strontium alumino silicate, AS= amorphous silica, FSG=fluoroalumino silicate glass, NFS= Nanofiller silica, TD= Titanium Dioxide, PFSC= prepolymerized filler of silica composite

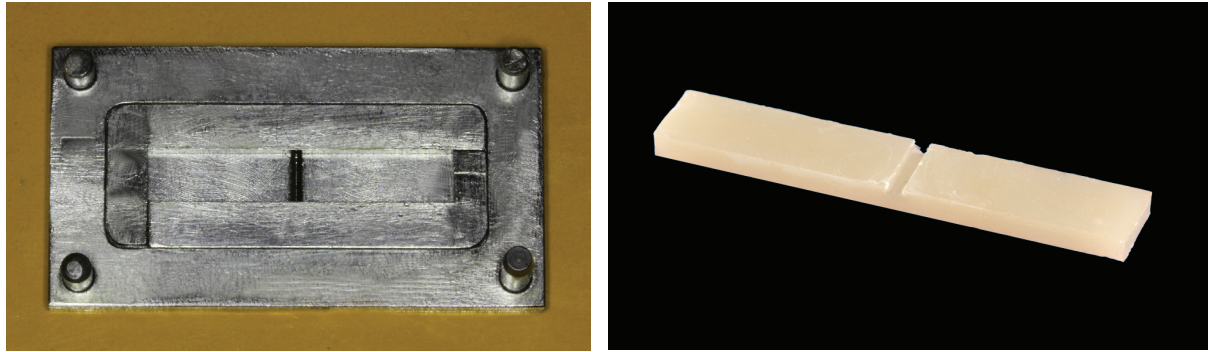


Figure 1: Brass aluminium mould and bar-shaped specimen

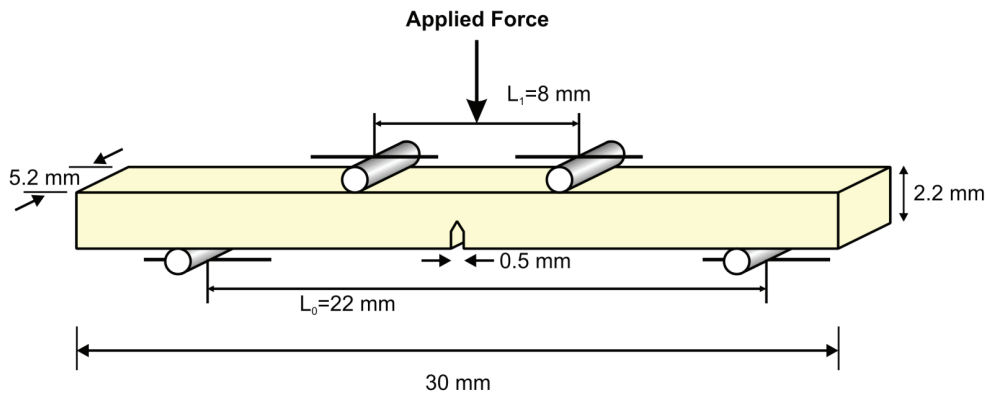


Figure 2: Specimen geometry for the determination of fracture toughness by the four-point bending test

from each material by filling the mould and pressing between transparent plastic strips and glass plates to extrude the excess material. The materials were cured according to the manufacturers' instructions for 40 seconds using a light-polymerizing.

LED unit with a wavelength range of 440-480 nm at an output of 1500mW/cm² (Radii plus LED, SDI, Bayswater, Vic, Australia). Each specimen was removed from the mould and light-cured on the opposite side for an additional 40 seconds. In order to obtain a flat surface, the edges of the specimens were gently ground manually with a circular motion with wet 1000-grit and then 1500-grit silicon carbide papers with brief rinsing in tap water between each grit. A new razor blade was used under hand pressure to create a sharp crack in the notch. Crack length (d) was measured and recorded for each specimen using a stereomicroscope (LV 150 Eclipse; Nikon, Japan) at 60x magnification. The specimens of each material were randomly divided into 3 groups of 8 and stored in distilled water at 37°C either for 24 hours or 21 days. Baseline measurement of K_{Ic} was performed after 24 hours of immersion. The specimens in the second group were kept in distilled water for 21 days. After 24 hours immersion in distilled water, the specimens in the third group (treatment), were bleached on one surface using Polanight 10% Carbamide Peroxide (SDI, Victoria, Australia), for a period of 21 days, temperature for 2 hours, then washed under tap water

and returned to distilled water.

The width, height and thickness of each specimen were measured using Dental Metal Gauge 04070 (Hygeia Dental Co.Ltd, Hong Kong, SAR China). Figure 2 demonstrates the specimen geometry for the determination of fracture toughness by the four-point bending test. The specimens were placed in the universal testing machine (Zwick/Roll Z020, Zwick GmbH & Co, Germany) using a four-point bending test jig, loaded at a cross-head speed of 0.05mm/min, and calibrated using the internal calibration.

The maximum load at specimen failure was recorded and the K_{Ic} (MPa.m^{0.5}) was calculated using the following formula:

$$K_{Ic} = \frac{L_{max}}{w\sqrt{h}} \cdot \frac{l_0 - l_1}{h} \cdot \frac{3 \cdot rM \sqrt{d/h}}{2 \cdot (1 - d/h)^{3/2}}$$

$$rM = 1.9887 - 1.326 \frac{d}{h} \cdot \frac{[3.49 - 0.68d/h + 1.35(d/h)^2]d/h(1 - d/h)}{(1 + d/h)^2}$$

L_{max} = maximum load

l_0 and l_1 = the outer and inner roll span

w and h = width and height of the specimens

d = length of the crack

Two-way analysis of variance (ANOVA) was performed to evaluate the interaction of materials and treatment. Then, one-way ANOVA was used for each material using Tukey's test to compare the effect of distilled water and bleaching agent individually. An alpha level of 0.05 was considered as statistically significant.

Table 2: Mean fracture toughness (MPa.m^{0.5}) and standard deviations (±) of the materials (n = 8)

Materials	24 Hours Distilled Water	21 Days Distilled Water	21 Days Carbamide Peroxide
Rok	3.4±(0.23) ^{Aa}	2.7 ±(0.1) ^{Ba}	2.5±(0.48) ^{Ba}
Vita-l-escence	3.00±(0.46) ^{Aa}	2.3±(0.6) ^{Ba}	3.07±(0.8) ^{Aa}
Estelite	3.1±(0.4) ^{Aa}	1.5±(0.1) ^{Bb}	1.9±(0.48) ^{Cb}

Means with the same upper-case letter in each row are not significantly different (p > 0.05). Means with the same lower-case letter in each column are not significantly different (p>0.05).

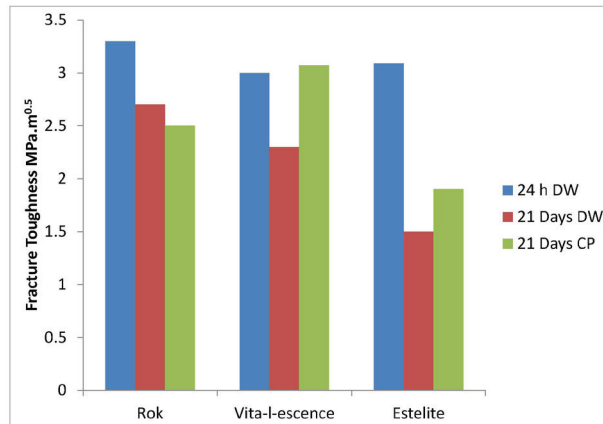


Figure 3: Fracture toughness (K_{1c}) mean values (MPa.m^{0.5}) versus time interval for all materials in distilled water (DW) with and without applying 10% carbamide peroxide (CP)

Results

Table 2 shows the mean values and standard deviations for K_{1c} of the tested resin composites in different times and media. The results has been graphically shown in Figure 3.

The results of two-way ANOVA showed a relationship between materials and treatment (P<0.001). After 24 hours of immersion in distilled water, Rok revealed the highest K_{1c} (3.4MPa.m^{0.5}) followed by Estelite (3.1MPa.m^{0.5}) and Vit-l-escence (3.00MPa.m^{0.5}) with no significant differences (P=0.085). Statistically significant differences were found between materials after 21 days either with or without applying the bleaching agent. Immersion in distilled water for 21 days was associated with a decrease in the fracture toughness of all materials in a significant level. In comparison with 24 hour immersion in distilled water, 21 days of treatment by carbamide peroxide led to a significant decrease on Rok and Estelite but a slight increase on Vit-l-escence. For each material, the difference between the groups is shown in Table 2.

Discussion

The outcome of this study rejected the null hypothesis, indicating that immersion in distilled water with and without applying 10% carbamide peroxide affected the fracture toughness of all resin composites in different

degrees. The effect of distilled water and bleaching agent on the K_{1c} (Table 2) was material dependent.

Measuring the fracture toughness of brittle materials using the 4-point bending test is achieved using flexure bars with a notch, at the tip of which a microscopic sized crack is created using a razor blade under hand pressure. Brittle materials are easier to fracture because they typically fail catastrophically. The origin of the failure is the point at which the stress is localized, i.e. the crack tip. Materials fracture when the stress intensity reaches a critical value, this is referred to as the fracture toughness of the material (K_{1c}) [19].

Fracture toughness (K_{1c}) is an intrinsic characteristic of materials describing resistance to crack propagation and more essentially determination of the critical role of residual stresses, and preexisting flaws. A resin composite with a homogenous structure will fail more slowly compared to a structure with the combination of flaw severity and stress demands which accumulate during manipulation and polymerization [16]. Filler’s type and size are shown to play an important role on the fracture toughness of resin composites [16].

As shown in Table 2 and graphically in Figure3, the effect of bleaching agent and distilled water was material-dependent. Vit-l-escence and Estelite lost their toughness significantly after 21 days of immersion in distilled water, but their toughness improved by applying 10% carbamide peroxide significantly. On the other hand, Rok with the highest value at baseline lost its toughness remarkably after 21 days of immersion

in distilled water and even slightly more with applying 10% carbamide peroxide. Therefore, the authors speculated that reduction of K_{Ic} after 21 days may be mainly due to aging in distilled water rather than treatment by 10% carbamide peroxide.

This result is in agreement with those of many other studies, which evaluated the fracture toughness of resin-based materials after aging in water for extended periods (one month or more) [7,16,20]. Our previous study [16] on the effect of immersion in distilled water on a resin composite and two PAM-RCs revealed that the K_{Ic} decreased as the time interval increased, with the highest decrease after 8 weeks of immersion. The reduction of fracture toughness due to aging could be attributed to many factors, including water sorption by the resin composite, which is dependent on the matrix resin, the filler and the properties of the interface between the matrix and the filler [21]. Different parameters such as storage time, pH of the storage media, generic types of composite, time of polymerization, and various chemicals can influence water sorption of resin composites [9,14]. Water sorption may cause degradation of the physical properties and decrease the life of dental materials by diverse effects such as dissolution, hydrolysis, expansion, plasticization, or microcrack formation and fatigue [22].

There are different reports regarding the influence of water sorption on the silane coupling agent.

Although only the polymer takes up water and there is minimal absorption of water by the filler particles, the interface between the filler particles and matrix provides a path for diffusion [22]. Water exposure may decrease the life of resin composites by silane hydrolysis and microcrack formation [12]. Excessive water uptake can promote the breakdown causing filler-matrix debonding [12]. The outcome of water absorption alters the mechanical properties of resin composites such as strength and hardness [23].

Most commercial resin composites contain dimethacrylates as an organic resin matrix and inorganic filler particles coated with methacrylate-based functional silane coupling agents to bond the resin to the filler [24]. The reduction in fracture toughness due to aging could also be accredited to the breakdown of the resin and the silane bond between the filler particles and the resin, associated with water sorption. The silane agent plays a critical role in forming an interfacial bridge that strongly binds the filler to the resin matrix, enhancing the mechanical properties of the composite and minimizing the plucking of the fillers from the matrix during clinical wear. A good silane bond provides a medium for stress distribution between adjacent particles and the polymer matrix as well as a hydrophobic environment that minimizes water absorption of the composite. On the other hand, it has been shown that poorly bonded filler

particles promote crack tip blunting and unstable crack propagation, while well-bonded filler particles promote the stable crack propagation [24].

The effect of bleaching agents on the physical properties of resin composites remains controversial. The application of 30-35% hydrogen peroxide did not affect the surface texture of the resin composites [25]. However, a slight, but statistically significant, increase in the surface roughness, and porosity of a micro-filled and hybrid resin composite was reported after application of 10-16% carbamide peroxide [26]. Turker et al. [27] found a reduction in micro hardness of resin composites after application of home-bleaching gels while the other group found an increase in hardness [28]. The differences among research papers may be due to the manner in which the hydrogen peroxide compounds were applied to the resin composite surface.

It is speculated that 15% carbamide peroxide immediately breaks down into one-third hydrogen peroxide (HP) and two-thirds urea on contact with tissue and saliva [29]. It is suggested that these peroxides may induce oxidative cleavage of polymer-chains. Thus if unreacted double bonds remain in a resin composite, they will be vulnerable parts of the polymer structure [29]. Furthermore, free radicals induced by the peroxides may affect the resin-filler-interface and cause debonding at this interface. Due to this effect, different amounts of filler in the materials tested in this study may account for the observed different effects of the bleaching agents [30].

In this study Rok and Estelite with filler volume of 67.7% and 63% respectively showed a higher value than Vit-l-escence with the lowest filler particle volume of 52 %. A previous research [20] also found a direct relationship between filler volume fractions and K_{Ic} of resin-based materials. However, Estelite and Rok showed a significant decrease of K_{Ic} after 21 days of applying carbamide peroxide while Vit-l-escence showed a slight increase.

Moreover, Vit-l-escence based on Bis-GMA alone showed lower K_{Ic} than Rok based on a blend of UDMA/TEGDMA/Bis-EMA and Estelite based on Bis-GMA/TEGDMA. It is reported that water sorption is influenced by the quantity of hydroxyl groups within the matrix, and creation of hydrogen bonds with water [31]. A ratio of Bis-GMA (70%)/TEGDMA (30%) mixture is suggested as the best ratio in terms of volumetric changes that accompany water uptake [32]. Composites based on TEGDMA alone exhibited higher diffusion coefficients than those based on a blend of UEDMA and TEGDMA [12]. A study results [32] reported that the uptake of water increased from 3 to 6% as the proportion of TEGDMA increased from 0 wt% to 1 wt%. It is preferable to use as little TEGDMA as that consistent with the practical need to use a monomer mixture with a sufficiently low viscosity [32].

Conclusion

Within the limitations of this study, the following conclusions were made:

After 24 hours of immersion in distilled water, Rok had the highest mean K_{1c} and Vit-l-escence the lowest with no significant differences. Immersion in distilled water for 21 days caused a significant decrease in K_{1c} values of all materials ($P < 0.05$). Treatment by carbamide peroxide was associated with a significant decrease on Rok and Estelite but a slight increase on Vit-l-escence. A direct relationship between filler volume fractions and K_{1c} of resin composites was found. The results of this study suggest that it may be prudent for clinicians to use those resin composites that their strength remains more stable over time.

Conflict of Interest: None declared.

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