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Review Article

Fracture Toughness of Resin Composites under Different Modes and Media: Review of Articles

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Abstract

This article aims to review various modes of fracture toughness of resin composites. Also, this study intends to review the papers on the fracture mode, namely "fractography", under scanning electron microscopy finding fracture initiation site, and the effect of filler content on the fracture toughness of resin composites. It will also review the effect of aging on the fracture toughness of resin composites in different media, mainly distilled water, and acidic environment. In the review performed on fracture toughness of resin composites we used "fracture toughness (K_{Ic})", aging AND fracture toughness, AND fractography" of resin composites as the search strategy. The outcome of the review revealed that most of the studies investigated fracture toughness of resin composites under Mode I and less under mode II. However, some others looked at the fracture toughness of dental resin composites under mixed-mode loading conditions. It was also found that fracture toughness studies performed on the same types of resin composites resulted in different values of K_{Ic}. The differences were related to the method of performance that requires different specimen geometries.

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Introduction

Fracture toughness is an inherent characteristic of a material that describes its ability to resist crack propagation [1]. The "plane strain fracture toughness", (K_{Ic}), is a measure for the crack resistance of a material. [2]. It is defined as the critical value of the stress intensity factor at a crack tip which produces catastrophic fast fracture [3]. K_{Ic} is an important measure of a material's properties, as it indicates the largest

amount of stress that a material can withstand prior to failure and represents the ability of a material to resist crack propagation from an existing flaw. Therefore, characterization of this property can help prevent devastating failures of resin composite restorations.

The main concern with restorative composites is to increase the fracture toughness, and consequently prolong their service life in the oral cavity while maintaining their aesthetic value. Although longevity and survival studies in posterior teeth continue to show that amalgam has a better track record than composite [4,5], a new formulation of resin composite is continually appearing on the market with improved mechanical properties [6].

Uunderstanding the failure mechanisms and the correlation between the laboratory strength tests and clinical behaviour of resin composites still need to be established to enhance their survival. The values of strength or failure load have been associated with the failure mode. Hence, the efforts are being made to find an appropriate method to reproduce the damage process occurring in service.

Due to the complexity of the forces that direct restorations to resist in the oral cavity, it is not easy to select a suitable method for testing fracture toughness of resin composites [7]. In the oral environment, dental restorations are subjected to continuous mechanical loads which lead to progressive degradation and crack propagation, resulting in catastrophic failure of the restorations [8]. Moreover, preexisting voids introduced during material processing, imperfect interfaces, and residual stresses will further increase the failure of the restorations in a period of time [2].

Most of the published work is concerned with mode I straight-line crack growth, and toughness characterization of various composites, which have been exposed to air, water, ethanol, and other environments. The objective of this study was to review some of the literature on the strength of different types of resin composites under numerous fracture toughness tests subjected to various media. To do so, the topic is divided into two sections. In Section 1.1, fracture mechanics in general and the way it can be applied to resin composites are briefly explained. In section 1.2, a review of different fracture toughness studies performed on resin composites, focusing on specimen geometry, filler content and sizes, and mode of fracture, is presented. In section 2, the effect of aging and storage media on the fracture toughness of the resin composites is presented. The results of in-vivo fracture toughness tests conducted on different types of commonly used resin composites are presented at the end.

Discussion

1.1 Fracture Mechanics

Fracture mechanics is an important tool in supporting and expecting the durability of materials. Fracture mechanics can be used to expect the rate at which a crack can reach a critical size in fatigue or by environmental influences, and can be used to determine the conditions under which a rapidly propagating crack can be arrested [9].

Fracture toughness testing is standardized by the American Society for Testing and Materials [10,11]. A test method that has been used extensively in the study of fracture properties of brittle materials is the Mode I, also referred to as the Brazilian disk test or diametral tensile test [12,13]. The procedure of fracture tough ness measurement involves creating a sharp crack tip. The crack tip condition is difficult to satisfy in brittle materials due to problems associated with growing a sharp crack normal to the applied load. Researchers have implemented various techniques to introduce sharp notches in brittle specimens including single-edge-notched beam, chevron notch, compact tension, and indentation hardness method. [14,15]. The study of fracture surface markings on brittle materials has been well documented. During failure, the crack front propagates through the material, creating fracture features known as the mirror, mist, and hackle (Figure 1).

The crack front initially produces a smooth mirror region. However, as the crack accelerates, it becomes more unstable, creating a dimpled surface known as mist. This instability eventually causes the crack to branch out, producing the rough hackle region. The hackle region is characterized by elongated markings that proceed in the direction of crack propagation [16]. Our recent study looked at the fracture pattern of resin

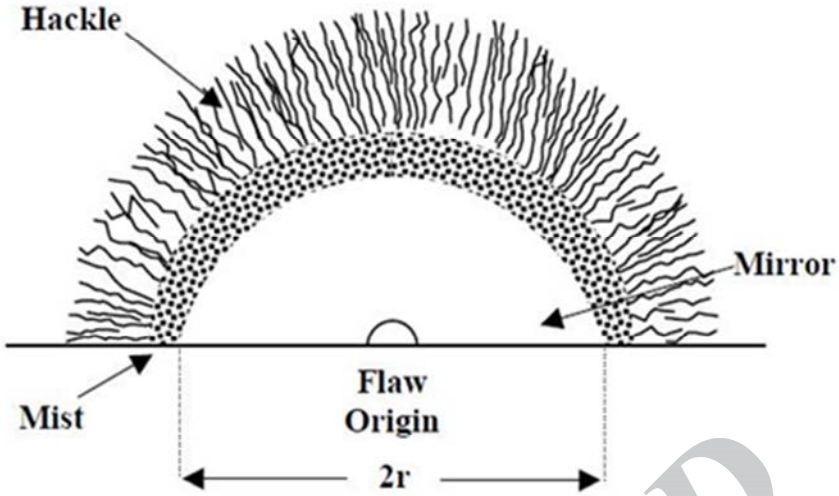

Figure 1: Schematic view showing brittle material surface features formed during failure [16]

composite, showing a typical pattern of the mirror, mist and hackle (Figure 2).

1.2. Modes of fracture in resin composites

A variety of fracture toughness testing methods has been used to evaluate the relative fracture toughness of resin composites. Those tests are classified as Mode I (tensile opening force), Mode II (shear opening

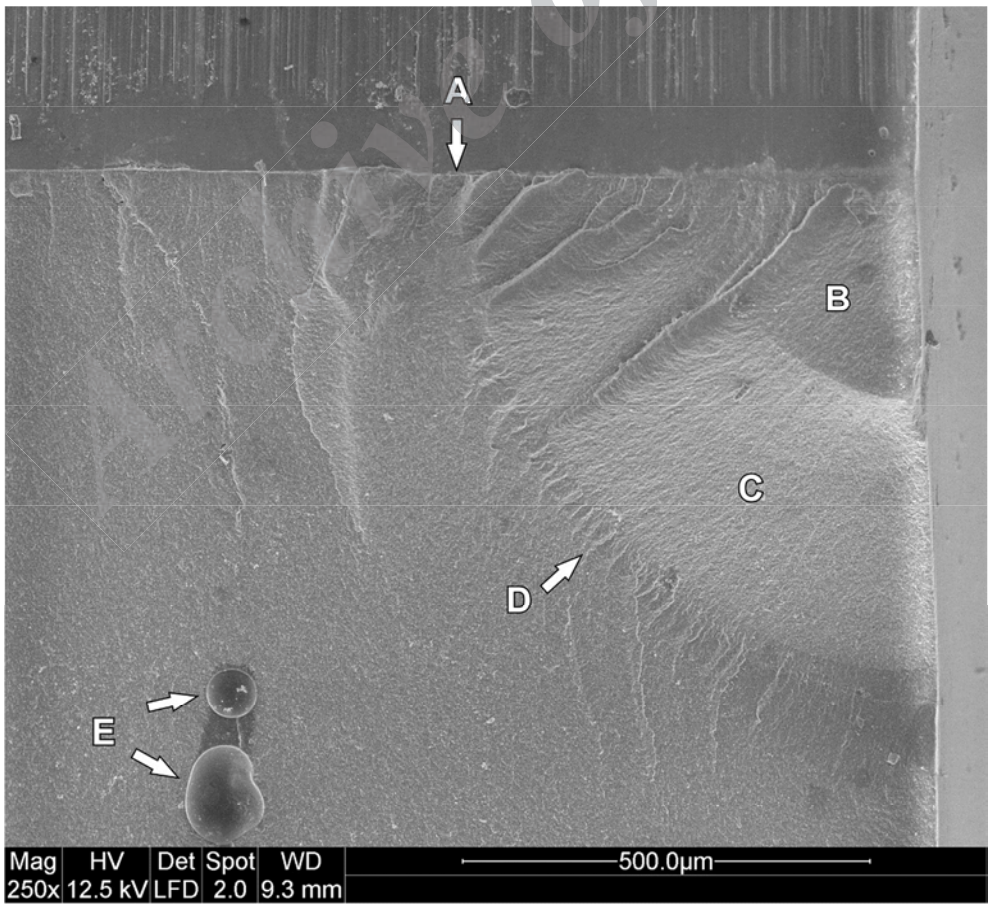

Figure 2: Compact tension fracture test of Esthet X showed "artificially introduced line notch interface" (A). Note the corner stress initiating fracture depicted by the location of the mirror region (B), mist region (C), hackle region (D), and voids (E)

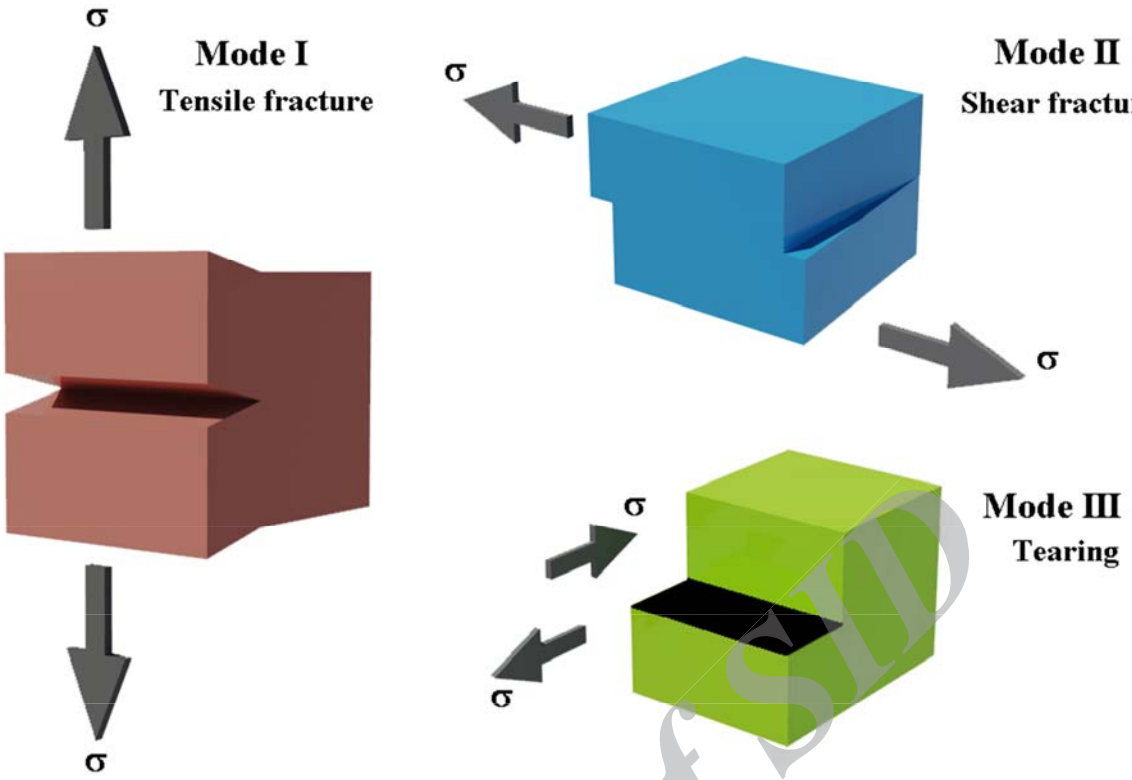

Figure 3: Schematic view of different modes of fracture toughness

force), and mode III is a mixture of I & II referred to as tear force (Figure 3). The Mode I fracture toughness (K_{Ic}) is the lowest stress at which catastrophic crack propagation will occur due to its tensile mode of opening [6]. It has been shown that [17] microstructural features are important in determining the strength and fracture toughness, whereas fatigue resistance is mainly related to the susceptibility of the matrix and the filler/matrix interface to mechanical and chemical degradation. Since surface fracture analysis only shows the region where the final failure occurs, many studies aimed to determine the degree of cracking in three dimensions in dental composites under various loading and environmental conditions. Two common fracture toughness tests for brittle materials including resin composites are 3-point and 4-point testing methods. Initially Drummond *et al*. [18] used 3-point test and it was continued by others [19-24]. In their study, Drummond *et al.* [18] found that the fractography image contrasts varied between samples due to reconstruction artifacts. One of the shortcomings of 3-point and 4-point tests was mentioned to be controlling the applied load because the load fluctuates while the sample is being examined; also, occasionally the specimen splinters during testing [18]. However, dental composites when placed within natural teeth are subject to radial as well as axial stresses, thereby introducing a three-dimensional (3D) compressive stress state [25,26]. Therefore, to replicate the loading that resin composites experience in the oral mouth condition, it is of relevance to examine resin composite restorations subjected to multiaxial compression loads, rather than uniaxial compression.

To overcome these issues of using uniaxial compression, a method of multiaxial compression loading was employed. Using a method described by Ma and Ravi-Chandar [27] to constitutively characterize materials under confined compression that the principle components of stress and strain could be determined. Outcome of this method may yield greater insight into the failure mechanisms of dental composites. This method also allows for better control of compressive load conditions, with the dental composite fabricated as cylindrical specimens [28].

Ravindranath *et al*. [29] evaluated the fracture in dental resin composite under mixed-mode loading conditions. They used diametral disk specimens 25 mm in diameter and 2 mm in thickness. Two methods

were used for generating initial cracks in the specimen. The first method involved machining a 3-mm notch in the center of the disk specimens, and then the notch tips were sharpened with a 0.2-mm-diameter jeweler's saw blade. In the second method for obtaining sharper crack tips, a three-way wedge was forced into a 3.17-mm hole drilled in the center of the specimen, resulting in sharp cracks starting from the notch tips. The maximum tensile stress (MTS) criterion was used to predict the fracture in dental resin composite under mixed-mode loading conditions. The loads at failure were used as input into a finite element model. After obtaining the stress field in the specimens by the finite element method, the mixed-mode stress intensity factors were calculated using an interaction energy integral method. Good agreement was obtained between the fracture envelope predicted by the MTS criterion and the experimental fracture toughness data. Hence, it can be concluded that it is only necessary to characterize the mode I fracture toughness to fully characterize the mixed-mode behavior of the dental resin composites that were considered in the present study [29].

Another study [30] determined fracture toughness of six commercially available nanofillers containing resin composites compared to a microhybrid and a microfilled material. The microfilled composite consistently showed the lowest values, and the microhybrid performed slightly better or in line with the nanohybrid and nanofilled materials. The study concluded that the filler size of the resin composites has a significant effect on its fracture toughness. Elbeshiri *et al*. [31] investigated the correlation between filler size, fracture toughness and voids. The percentage of voids and fracture toughness data was analyzed, showing that filler size was strongly correlated to the voids percentages but it had no effect on fracture toughness. Seven model resin composites and one commercial were used in the study. A single edge notch mould was used to prepare the samples. A selected area of 1mm below and above the notch was scanned with micro CT and then the percentage of voids was calculated [31].

The fracture toughness of a large number of dental restorative material categories was analyzed [32]. The fracture toughness (K_{IC}) of 69 restorative materials belonging to ten material categories was measured by means of the single-edge notched-beam method after storage for 24 h in distilled water. The materials were categorized into micro-hybrid, nanofilled, microfilled, packable, ormocer-based and flowable resin-based composites (RBC), compomers and flowable compomers, as well as glass ionomer cements (GIC), and resin-modified GIC. Large variations were found between the tested materials within a material category. The lowest fracture toughness was observed in the GIC group, followed by the microfilled RBCs, resin-modified GIC, and flowable compomers, which do not differ significantly among each other as a material group. The ormocer-based, packable, and micro-hybrid RBCs performed statistically similar, reaching the highest fracture toughness values. The correlation between K_{IC} and filler volume and respective filler weight was low. K_{IC} increased with the volume fraction of fillers until a critical value of 57%. Above this value, K_{IC} decreased slightly. The authors recommended that due to the very large variability of the fracture toughness within a material type, the selection of a suitable restorative material should have not been done with respect to a specific material category, especially in stress-bearing areas, but by considering the individual measured material properties [32]. Another study [33] characterized the microstructure and composition of two different composites, and determined their influence on the physical properties and fracture behavior. The microstructure and composition of a microhybrid (Filtek Z250™-Z2) and a nanofill (Filtek Supreme™-SU) composite were analyzed using scanning electron microscopy (SEM) and electron dispersive spectroscopy (EDS). Filler wt% was determined by thermogravimetric analysis. Fractographic analysis (FA) was performed to determine the fracture origin (c) for calculation of fracture toughness (K_{Ic}), and these results were compared to those from the single edge notch beam (SENB) method. Results revealed that the microstructure did not influence the fracture behavior and the structural reliability of these highly filled composites. Fractographic analysis was shown to be a reliable method for determining the K_{Ic} of the composites [33].

A recent study [34] compared the fracture toughness (K_{Ic}) obtained from the single edge V-notched

beam (SEVNB) and the fractographic analysis (FTA) of a glass-infiltrated and a zirconia ceramic. All specimens were loaded to the fracture, using a universal testing machine at a crosshead speed of 0.5-1 mm/min. The mean K_{Ic} of glass-infiltrated ceramic obtained from SEVNB method was significantly lower than that obtained from FTA method. The author concluded that the differences in the K_{Ic} values could be a result of the differences in the characteristics of fracture initiating flaws of these two methods [34].

In 2014, Ornaghi *et al.* [35] verified the influence of filler size distributions on fracture toughness (K_{Ic}), initial fracture strength (IFS) and cyclic fatigue resistance (CFR) of experimental resin composites. Four composites were prepared with the same inorganic content (78 wt%), in which 67 wt% was constituted by glass particles with diameters of 0.5,0.9,1.2, and 1.9 μm. K_{Ic} of the composites was determined by the single-edge notched beam (SENB) method. To evaluate the IFS and the CFR, a biaxial bending test configuration was used. The CFR was determined under cyclic loading for 10(5) cycles using the 'staircase' approach. The fracture surfaces of IFS and CFR specimens were analyzed under scanning electron microscope (SEM). There was a positive linear correlation between diameter and KIc. SEM images showed the morphology with brittle fracture patterns for the surfaces of IFS specimens and a more smooth fracture surface for CFR specimens [35].

Watanable *et al*. [3] investigated the Mode I and II fracture toughness values of resin composites used for the restorations of the anterior teeth by the Brazilian disk test method. The highest mean Mode I and II fracture toughness values were found in hybrid and nano-hybrid resin composites compared to those of micro-filled resin composites. This suggests that the micro-filled resin composites should be used for non-stress bearing areas [3]. The linear elastic material properties of direct dental resin composites were measured and then correlated with their fatigue strength under cyclic loading [36]. Bar specimens of twelve resin composites were produced for elastic modulus in both 3-point and 4-point bending, using the same specimen geometry. We observed the fracture surface and fracture profiles, using a scanning electron microscope in order to evaluate the respective fracture mechanisms according to the two different loading conditions. Materials were ranked differently according to the tested parameters. Crack path in both loading conditions was mainly inter-particle, with the crack propagating mainly within the matrix phase for fatigued specimens and eventually through the filler/matrix interface for statically loaded specimens [36].

Because fracture toughness is a characteristic property of a material, its value should be independent of the mode of measurement. Determination of K_{Ic} is technically sensitive, and the values obtained and subsequent rankings may differ depending on the techniques and procedure used. Many studies have reported widely different values of fracture toughness for the same type of materials [23,24,37]. The most likely reason for these differences is variation in specimen fabrication, since fracture toughness reflects the ability of the cracks to propagate through the material, and such ability is dependent on the defect density. However, Fujishima *et al*. [1] concluded that although the double torsion test was the most technique-sensitive one among the four methods of mode I, it provided the most information about crack initiation and propagation and may be the most appropriate technique.

2.1. Effect of aging and storage media on the fracture toughness of resin composites

A laboratory study [21] compared the fracture toughness (K_{Ic}) of tooth-coloured restorative materials based on a four-point bending, assessing the effect of distilled water and a resin surface sealant (G-Coat Plus) on the resistance of the materials to fracture. They found a significant difference among most of the materials. Hybrid resin composite had the highest and glass ionomer the lowest mean values. Immersion in distilled water for the resin composite and polyacid-modified resin composites caused a significant decrease in K_{Ic} as the time interval increased. For glass-ionomer cements, K_{Ic} decreased significantly after 4 weeks, and after 8 weeks immersion slightly increased [21]. The increase in K_{Ic} from 4 to 8 weeks is difficult to explain. It could be the result of a change from a brittle failure to a plastic failure, although other factors such as delayed polymerisation or setting stress relaxation may be considered.

It has been shown that [24,37] fracture toughness of the resin composites was affected by the bleaching agent and distilled water, in which fracture toughness values of nanofilled resin composites were decreased due to aging and application of bleaching agent. The study [24] determined the effect of immersion time in distilled water, with and without exposure to 10% carbamide peroxide on three types of resin composites, i.e. hybrid, microhybrid, and nanofilled by employing short rod design fracture toughness test. Study group specimens were bleached for 21 days, 2 hours a day. A significant relationship was found between material and time; after 24 h of immersion in distilled water, hybrid composite revealed the highest K_{Ic} followed by microhybrid and nanofilled composites. In comparison with hybrid and nanofilled, fracture toughness of microhybrid was increased due to aging and application of bleaching agent [24]. On the other hand, the bleaching agent significantly decreased the fracture toughness values of nanofilled resin composited; this is in agreement with the results of other studies [37]. They [37] found that the application of bleaching agents did not significantly change the fracture toughness values of all nanofilled resin composites tested except for Filtek Supreme Plus. They evaluated the effect of four concentrations of bleaching agents: Opalescence PF 10%, 20%, 35%, and 45% on four nanofilled resin composites for 14 days. The specimens were subjected to a three-point bending test with a crosshead speed of 0.2 mm per minute [37].

Another study [23] assessed the effect of distilled water and a home bleaching agent on the fracture toughness (K_{Ic}) of resin composites under four-point bending test. Seventy-two bar-shaped specimens were prepared from three types of resin composite materials: Hybrid, nanohybrid, and microhybrid. 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 for 2 hours daily. For each material, a total of 24 disc-shaped specimens were prepared and loaded after each time interval 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. Statistical analysis showed a significant relationship between materials and treatment. K_{Ic} did not significantly affect the materials after 24 hours immersion in water; hybrid revealed the highest value followed by nanohybrid and microhybrid, respectively. The bleaching agent significantly decreased the K_{Ic} values of nanohybrid and hybrid while it did not affect that of microhybrid. Immersion in distilled water for all resin composites caused a significant decrease in K_{Ic}. The fracture toughness of the resin composites was affected by the bleaching agent and 21day immersion in distilled water [23].

Lactic acid has been shown to affect the fracture toughness (K_{Ic}) of hybrid and nanohybrid resin composites under tensile loading after three months of immersion. Immersion in either distilled water or lactic acid significantly decreased the fracture toughness of almost all materials as time interval increased [38]. The results are in agreement with those of many other studies assessing the fracture toughness of resin-based materials after aging in water for extended periods of one or more months [39-41,30].

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 filler [42]. Water sorption by polymers is a diffusion process and most water sorption occurs into the resin matrix [43,44]. The greater the resin content, the more water is absorbed [43,45-47]. Water sorption causes softening of the resin matrix, leading to plasticization and a gradual degradation of the material [48]. Excessive water uptake can promote the breakdown, causing filler-matrix debonding [49]. On the other hand, the effect of water sorption causes softening of the composite by penetration into the matrix followed by leaching out of unreacted monomer, degradation and leaching of filler components [50,51]. Water exposure may decrease the life of resin composites by silane hydrolysis and microcrack formation [49]. Hence, the outcome of water sorption may alter the strength and fracture toughness of resin-based materials.

However, once the network is saturated with water, the structure is likely to stabilize and there may be no further reduction in fracture toughness. It has been

reported that aging in water led to a significant decrease in the fracture toughness of resin composites in the first 6 m with minimal changes from 6-24 m [39,52].

Other theories as to the cause of the degradation to the dental resin include the formation of microcracks through repeated sorption/desorption cycles leading to hydrolytic degradation of the polymer [53,54]. Some other studies [55,56] on the comparison of fracture toughness of glass ionomer cements and resin composite revealed that fracture toughness of these materials was significantly affected by testing environment, aging and cyclic loading. In Bapna *et al*.'s study, [55], two sets of specimens were evaluated: a set of controls (at 37°C and 95% humidity) and a set aged for 9 months at 37°C in water. The specimens were tested in static loading in air and water, and cyclic loaded in water. Fracture toughness was determined by three-point loading using a material testing system. Wet static fracture toughness did not change on aging, and occasionally increased. Cyclic fracture toughness was also lower with all materials except a hybrid glass ionomer without aging. Deterioration in properties was related to relative amounts of glass ionomer vs. the resin component. The lower the resin component of the material, the lower the mechanical properties. The influence of these parameters is of importance in considering their clinical durability in the oral environment [55].

Knobloch *et al*. [56] showed a significant increase in fracture toughness values of resin-based luting cements after 24 hours and 7 days storage in distilled water. This study determined the fracture toughness of resin cements by preparing minicompact test specimens with introduced precracks (in tensile mode).

Nowadays, the main concern of dental clinicians is fracture toughness and clinical survival of posterior resin composite restoratives. Clinical data has suggested that microfills are more susceptible to bulk fracture [57]. Studies have indicated a range of failure attributed to bulk fracture of the composite from very low [58] to 7% [59] and 32% [60]. Many studies have been conducted indicating that packable composites on average have a fracture rate of 8% after 1.5 years [61], 7% after 2 years [62], 7–14% after 2 years [63], and 19% after 3.5 years for a packable composite. For a hybrid composite the following fracture rate has been reported: 8% after 3.5 years, 14% after 8 years [64], and 35% after 17 years [65]. Cusp fracture as a source of failure has been reported to be the same for either amalgam or composite restorations [66].

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