

EFFECT OF AGING ON THE FLEXURAL STRENGTH AND FRACTURE TOUGHNESS OF A FIBER REINFORCED COMPOSITE RESIN VERSUS TWO NANOHYBRID COMPOSITE RESINS

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ABSTRACT

INTRODUCTION: It is known that posterior composite restorations have high failure rates and high frequency of replacement as shown by studies. This may be attributed to the inability of conventional fillers to withstand the forces of mastication in the posterior region. New methods of reinforcement such as glass fibers are being used to increase the mechanical properties of dental composites.

OBJECTIVES: was to compare the effect of aging in distilled water at 37°C for 1 day, 3 months and 6 months on the flexural strength and fracture toughness of a fiber reinforced composite (EverX posterior, GC, Europe), a nano-hybrid ceramic filled composite (IPS Empress Direct, Ivoclar Vivadent, Lieschtenstein) and a nano-hybrid zirconia filled composite (Z250 XT, 3M ESPE, USA).

MATERIALS AND METHODS: For each test, twenty-one specimens were fabricated from each of the three composites and were then subdivided into three subgroups of seven specimens each according to the aging period in distilled water. After each aging period, the specimens were fractured in a Universal testing machine and the results were analyzed using ANOVA and post hoc test (Fisher's LSD) at $p < 0.05$ significance level. Following each test, the fractured surfaces of the 6 months aged specimens were examined using SEM.

RESULTS: The flexural strength and fracture toughness of the fiber-reinforced composite was the highest with a statistical significance in the three aging periods followed by the nano-hybrid zirconia filled composite and the least was the nano-hybrid ceramic filled composite. SEM imaging findings were consistent with the results.

CONCLUSIONS: The fiber-reinforced composite had the highest flexural strength and fracture toughness after each of the three aging periods. Although aging in water decreased the mechanical properties of the fiber-reinforced composite, it still remained higher than the two nano-hybrid composites which ensures its ability to withstand forces of mastication in the posterior region.

KEYWORDS: Fiber-reinforced, aging, flexural strength, fracture toughness, nano-hybrid.

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INTRODUCTION

Composite restorative materials represent a success of modern biomaterials research, since they replace biological tissue in both appearance and function (1). Their indications have been extended to direct anterior and posterior fillings, indirect inlays, onlays, veneers, crowns and partial fixed bridges (2).

Composite resin is composed of three distinct phases, each with its own role in dictating material properties: resin matrix, fillers, and the filler-resin interface (1). The filler type, shape and amount, as well as the efficient coupling of fillers and resin matrix, contribute to the material performance. Properties such as compressive strength, flexural strength, hardness and elastic modulus improve as the filler content increases (3).

According to Van Noort, it is possible to classify dental resin composites according to filler size into: macro-filled, micro-filled, hybrid, small particle hybrid and nano-filled resin dental composites (4). Nanocomposites are the premises of new materials that can be applied in many fields due to their improved mechanical properties, light weight, and light-conducting properties (5). Such materials are available as nanofill types, containing both discrete nanomer and nanocluster particles, and as nano-hybrid compounds containing milled glass fillers and discrete nanoparticles (40-50 nm). Nanocomposites are claimed to combine the good mechanical strength of the hybrids and the superior polish of the microfills (6).

Short fiber reinforced composite was introduced as a dental restorative composite resin. The composite resin is intended to be used in high stress bearing areas especially in molars. The results of the laboratory mechanical tests revealed substantial improvements in the load bearing capacity, the flexural strength and fracture toughness of dental composite resin reinforced with short E-glass fiber fillers in comparison with conventional particulate filler restorative composite resin (7).

The function of bulk short fiber composite substructure is based on supporting the surface particulate filler composite layer and working as crack stopper layer. Reinforcing effect of the fiber fillers is based on stress transfer from polymer matrix to fibers but also behavior of individual fiber as a crack stopper (8).

It has been reported that damage to composite materials may result from deterioration of the matrix and fillers or is due to mechanical and environmental loads, interfacial debonding, microcracking or filler particle fracture, which may reduce the survival probability of composite restorations in vivo (9).

When comparing the aging behavior of different dental materials, the clinical aging process is most commonly simulated in vitro, using defined artificial aging protocols (10). One of the laboratory tests most used to evaluate the mechanical behavior of resin composites is the flexural strength test. It simulates the complex forces that develop in areas of stress concentration, since it induces compressive

and tensile forces simultaneously, close to and opposite from the point of loading, respectively (11).

Dental materials researchers regard the fracture toughness of a material as a more accurate predictor than traditional compressive and tensile testing of how a material will perform under various occlusal and masticatory stresses (12). Taken together both flexural strength and fracture toughness determine the bulk characteristics, as opposed to a surface characteristic, of the resin composite material (13).

The hypothesis to be tested in this study is that there will be a difference in the fracture toughness and flexural strength of a fiber-reinforced composite, a nano-hybrid ceramic filled composite and a nano-hybrid zirconia filled composite after aging in distilled water for different periods of time.

MATERIALS AND METHODS

Three different types of composites with different types of fillers were used, Group I: a fiber-reinforced composite (EverX Posterior, GC, Europe), Group II: a nano-hybrid ceramic filled composite (IPS Empress Direct, Ivoclar Vivadent, Lieschtenstein) and Group III: a nano-hybrid zirconia filled composite (Z250 XT, 3M ESPE, USA). The compositions of the three composite materials are listed in (Table 1).

Table 1: The composition of the three composite materials used in the study.

Composite	EverX Posterior	IPS Empress Direct	Z250 XT
Manufacturer	GC Europe	Ivoclar Vivadent	3M ESPE
Type	Fiber-Reinforced composite	Nano-hybrid ceramic filled composite	Nano-hybrid zirconia filled Composite
Resin Matrix	Bis-GMA. TEGDMA. PMMA.	Bis-GMA. UDMA.	Bis-GMA UDMA TEGDMA Bis-EMA PEGDMA
Fillers	<ul style="list-style-type: none"> E-glass fibers length (1-2mm). diameter (17 μm) Barium borosilicate glass (0.1-2.2 μm). 	Barium Al-fluorosilicate glass. (0.7 μm) Prepolymer (1-10 μm). Ytterbium trifluoride (100 nm). Spherical mixed oxide (150 nm).	Surface-modified zirconia/silica (≤3 μm). <ul style="list-style-type: none"> Non-agglomerated/non-aggregated surface-modified silica (20 nm).
Filler loading (by weight)	77%	79 %	82 %
Filler loading (by volume)	53.6%	59%	68%

A- Flexural strength test

A total of sixty-three specimens were prepared for this test. Twenty-one specimens were prepared from each type of composite under investigation. The specimens were

fabricated according to ISO standard 4049/2000 (14) using a teflon split mold (15) with dimensions 25x2x2 mm (length x width x height) (11). A teflon split mold was used to ensure that the composite material will not stick to it and to ensure that no force was required to remove the cured specimens from the mold to prevent any internal or external stresses.

Composite material was inserted in bulk using compules and a gun for ease of insertion. Composite was covered with a celluloid strip on top of which a glass slide was placed to ensure a smooth surface. Photoactivation was carried out with three non-overlapping 20 seconds exposures using a LED curing unit (Elipar S10, 3M ESPE, USA) of 1200 mW/cm² light intensity and 10 mm tip diameter to cover the entire length of the specimen in three exposures. The mold was clearly marked to avoid overlapping of the exposures. After photoactivation, the top portion of the specimen was identified with a marker and excess material was removed with a scalpel blade number 11 (11).

The specimens of each group were subdivided into three subgroups of seven specimens each according to the aging time in distilled water and stored in glass beakers in the incubator (MLW BST 5020, Germany) at 37°C for 1 day as subgroup (a), for 3 months as subgroup (b) and for 6 months as subgroup (c).

After each aging period, each specimen was dried and aligned horizontally in the lower platform of the universal testing machine (Comten Industries, USA) with the light cured side of the specimen, identified with a marker, facing upwards.

The load was applied to the central part of the specimen with a knife-edge indenter with a span of 20 mm between the supports at a crosshead speed of 0.5mm/sec till it fractured. The maximum fracture load (L in Newtons) of each specimen was recorded, and the flexural strength (F_s) in MPa, was calculated as follows (11):

$$F_s = \frac{3LD}{2wh^2}$$

Where: L=Maximum fracture load in Newtons, D=Distance between the supports (20 mm), w=Specimen width (2 mm), h=Specimen height (2 mm).

B- Fracture toughness test:

A total of sixty-three specimens were prepared for this test. Twenty-one specimens were prepared from each type of composite under investigation. Specimens were prepared according to the American Society for Testing Materials (ASTM) guidelines for the single-edge notched beam specimen (Standard E-399) (16) using a custom made rectangular teflon split mold (17) and using a sharp stainless steel razor blade to produce a centrally placed notch. A teflon split mold was used to ensure that the composite material will not stick to it and to ensure that no force was required to remove the cured specimens from the mold to prevent any internal or external stresses.

The dimensions of the specimens were 5 x 2.5 x 20 mm (width x thickness x length) with a 2.5 mm notch on one side (18). Composite was inserted into the mold in two increments, each increment covering the entire length of the mold. Photoactivation was done for the first increment using a LED curing unit (Elipar S10, 3M ESPE, USA) in two non-overlapping exposures for 20 seconds each. The second increment was inserted into the mold and then the razor blade was put into the composite to produce a centrally placed notch. The top layer was covered with a celluloid strip on top of which a microscopic glass slide was placed to ensure a smooth surface

and then light cured. Excess material was removed with a scalpel blade number 11.

The specimens of each group were subdivided into three subgroups of seven specimens each according to the aging time in distilled water and stored in glass beakers in the incubator (MLW BST 5020, Germany) at 37°C for 1 day as subgroup (a), for 3 months as subgroup (b) and for 6 months as subgroup (c).

After each storage period, each specimen was dried and placed horizontally in the lower platform of the universal testing machine (Comten Industries, USA) with the notch facing downwards. The load was applied to the centre of the specimen with a knife-edge indenter with a span of 15mm between the supports at a crosshead speed of 0.5mm/sec till it fractured. Visual examination of the fractured parts was performed to ensure that the fracture plane was through the notch and that it was perpendicular to the vertical and horizontal planes through the center of the specimens (19).

Fracture toughness (K_{IC}) in $MPa.m^{1/2}$ was calculated according to the equation (19):

$$K_{IC} = \left(\frac{PL}{bw^{1.5}} \right) f \left(\frac{a}{w} \right)$$

Where,

$$f \left(\frac{a}{w} \right) =$$

$$\frac{3}{\alpha} \left(\frac{a}{w} \right)^{0.5} \left\{ 1.99 - \left(\frac{a}{w} \right) \left[1 - \frac{a}{w} \right] \left[2.15 - 3.93 \frac{a}{w} + 2.7 \left(\frac{a}{w} \right)^2 \right] \right\}$$

$$\text{and } \alpha = 2(1 + 2a/w)(1 - a/w)^{1.5}$$

Where,

k_{IC} = stress intensity factor, P= load at fracture, L = span distance between the supports, w= width of the specimen, b= thickness of the specimen, a= crack length.

Scanning electron microscope (SEM) examination

For both flexural strength and fracture toughness tests, after the aging period of 6 months, a specimen from each of the three composites was randomly selected and the fractured surfaces were viewed using the scanning electron microscope (JEOL JSM-5300, USA). The specimens were mounted on stubs, gold sputtered and viewed at a magnification of (1000x). SEM images of the fractured surfaces were obtained with an accelerating voltage of 15 kV.

STATISTICAL ANALYSIS

Calculation of flexural strength and fracture toughness using the mathematical equations was done and descriptive statistics were calculated as means and standard deviations. Comparison of mean flexural strength and mean fracture toughness at different aging periods was done using analysis of variance (ANOVA) at $p < 0.05$ significance level followed by Fisher's LSD test for pair wise multiple comparisons. Statistical analysis was done using SPSS (Statistical Package for Social Science, SPSS Inc., Chicago, IL, USA) program version 16.0.

RESULTS

A- Flexural strength

Statistical analysis showed significant difference between the flexural strength of the three composite materials at the different aging periods. The results are summarized in (Table 2) and represented graphically using simple bar chart (Figure 1).

Flexural strength of the fiber-reinforced composite was the highest in all the three aging periods with a statistical significant difference compared to the two nano-hybrid composites. The least was related to the nano-hybrid ceramic filled composite with a flexural strength half that of the fiber-reinforced composite at the different aging periods.

For the fiber- reinforced composite the flexural strength significantly decreased by about 17% after 3 months aging, from $(172.67 \pm 21.34 \text{ MPa})$ to $(142.47 \pm 14.97 \text{ MPa})$ and 23% after 6 months aging, from $(172.67 \pm 21.34 \text{ MPa})$ to $(123.87 \pm 9.25 \text{ MPa})$. For the nano-hybrid ceramic filled composite, the flexural strength significantly decreased by about 31 % after 3 months aging, from $(108.80 \pm 3.65 \text{ MPa})$ to $(79.85 \pm 11.07 \text{ MPa})$ and 35% after 6 months aging, from $(108.80 \pm 3.65 \text{ MPa})$ to $(69.31 \pm 5.65 \text{ MPa})$. For the nano-hybrid zirconia filled composite, the flexural strength significantly decreased by about 33% after 3 months aging, from $(157.34 \pm 5.89 \text{ MPa})$ to $(106.04 \pm 3.41 \text{ MPa})$ and 40 % after 6 months aging, from $(157.34 \pm 5.89 \text{ MPa})$ to $(92.57 \pm 3.29 \text{ MPa})$ but still remained at higher values than the nano-hybrid ceramic filled composite.

Table 2: Flexural strength results of the three composite materials at the three aging periods.

Flexural strength test (MPa)	EverX Posterior (n= 7)	IPS Empress Direct (n= 7)	Z 250 XT (n= 7)	F test value
1 Day				
Mean ± SD.	172.67 ± 21.34	108.80 ± 3.65	157.34 ± 5.89	46.383*
Significance between groups	$p_1 < 0.001^*$, $p_2 = 0.040^*$, $p_3 < 0.001^*$			
3 Months				
Mean ± SD.	142.47 ± 14.97	79.85 ± 11.07	106.04 ± 3.41	57.990*
Significance between groups	$p_1 < 0.001^*$, $p_2 < 0.001^*$, $p_3 < 0.001^*$			
6 Months				
Mean ± SD.	123.87 ± 9.25	69.31 ± 5.65	92.57 ± 3.29	122.638*
Significance between groups	$p_1 < 0.001^*$, $p_2 < 0.001^*$, $p_3 < 0.001^*$			
Significance between aging periods				
EverX Posterior	$p_4 = 0.002^*$, $p_5 < 0.001^*$, $p_6 = 0.043^*$			16.651*
IPS Empress Direct	$p_4 < 0.001^*$, $p_5 < 0.001^*$, $p_6 = 0.017^*$			52.337*
Z 250 XT	$p_4 < 0.001^*$, $p_5 < 0.001^*$, $p_6 < 0.001^*$			428.972*

p_1 : p value for comparing between EverX Posterior and IPS Empress Direct

p_2 : p value for comparing between Everx Posterior and Z 250 XT

p_3 : p value for comparing between IPS Empress Direct and Z 250 XT

p_4 : p value for comparing between 1 day and 3 months

p_5 : p value for comparing between 1 day and 6 months

p_6 : p value for comparing between 3 months and 6 months

*: Statistically significant at $p \leq 0.05$

Flexural strength Scanning electron microscope (SEM) imaging results

After aging in distilled water for 6 months, the scanning electron microscope images of the flexural strength

specimens' fractured surface revealed cohesive fractures in the resin matrix. In case of the fiber-reinforced composite, the fracture goes around the fibers and the fibers remained intact (Figure 2A) while in case of the nano-hybrid ceramic filled composite and the nano-hybrid zirconia filled composite the fracture goes through the resin matrix and the fillers (Figure 2B and 2C).

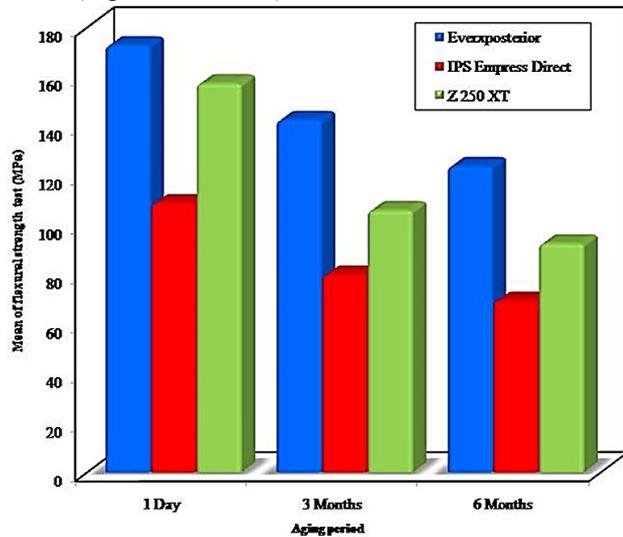


Figure 1: Comparison between the different composite materials in the same aging period and its effect on the flexural strength.

B- Fracture toughness

Statistical analysis showed significant difference between the fracture toughness of the three composite materials at the different aging periods. The results are summarized in (Table 3) and represented graphically using simple bar chart (Figure 3).

Fracture toughness of the fiber-reinforced composite was the highest in all the three aging periods with a statistical significant difference compared to the two nano-hybrid composites. The least was the nano-hybrid ceramic filled composite. The fracture toughness of the fiber-reinforced composite was about 4 times that of the nano-hybrid ceramic filled composite and 3 times that of the nano-hybrid zirconia filled composite at the different aging periods.

For the fiber-reinforced composite, the fracture toughness significantly decreased by about 37% after 3 months aging, from $(5.21 \pm 0.79 \text{ MPa}\cdot\text{m}^{1/2})$ to $(3.23 \pm 0.19 \text{ MPa}\cdot\text{m}^{1/2})$ and 42% after 6 months aging, from $(5.21 \pm 0.79 \text{ MPa}\cdot\text{m}^{1/2})$ to $(2.98 \pm 0.27 \text{ MPa}\cdot\text{m}^{1/2})$. For the nano-hybrid ceramic filled composite, the fracture toughness significantly decreased by about 35% after 3 months aging, from $(1.19 \pm 0.04 \text{ MPa}\cdot\text{m}^{1/2})$ to $(0.87 \pm 0.16 \text{ MPa}\cdot\text{m}^{1/2})$ and 50% after 6 months aging, from $(1.19 \pm 0.04 \text{ MPa}\cdot\text{m}^{1/2})$ to $(0.57 \pm 0.12 \text{ MPa}\cdot\text{m}^{1/2})$. For the nano-hybrid zirconia filled composite, the fracture toughness decreased by about 16% after 3 months aging, from $(1.27 \pm 0.18 \text{ MPa}\cdot\text{m}^{1/2})$ to $(1.11 \pm 0.17 \text{ MPa}\cdot\text{m}^{1/2})$ and 26 % after 6 months aging, from $(1.27 \pm 0.18 \text{ MPa}\cdot\text{m}^{1/2})$ to $(0.93 \pm 0.15 \text{ MPa}\cdot\text{m}^{1/2})$ but still remained at lower values than the fiber-reinforced composite.

Fracture toughness Scanning electron microscope (SEM) imaging results

After aging in distilled water for 6 months, the scanning electron microscope images of the fracture toughness specimens' fractured surface revealed that the fibers in the fiber-reinforced composite acted as micro-crack deflectors (Figure 4A) while the ceramic fillers in the nano-hybrid ceramic filled composite and the zirconia fillers in the nano-hybrid zirconia filled composite (Z250 XT, 3M ESPE,

USA) failed to act as micro-crack deflectors (Figure 4B and 4C).

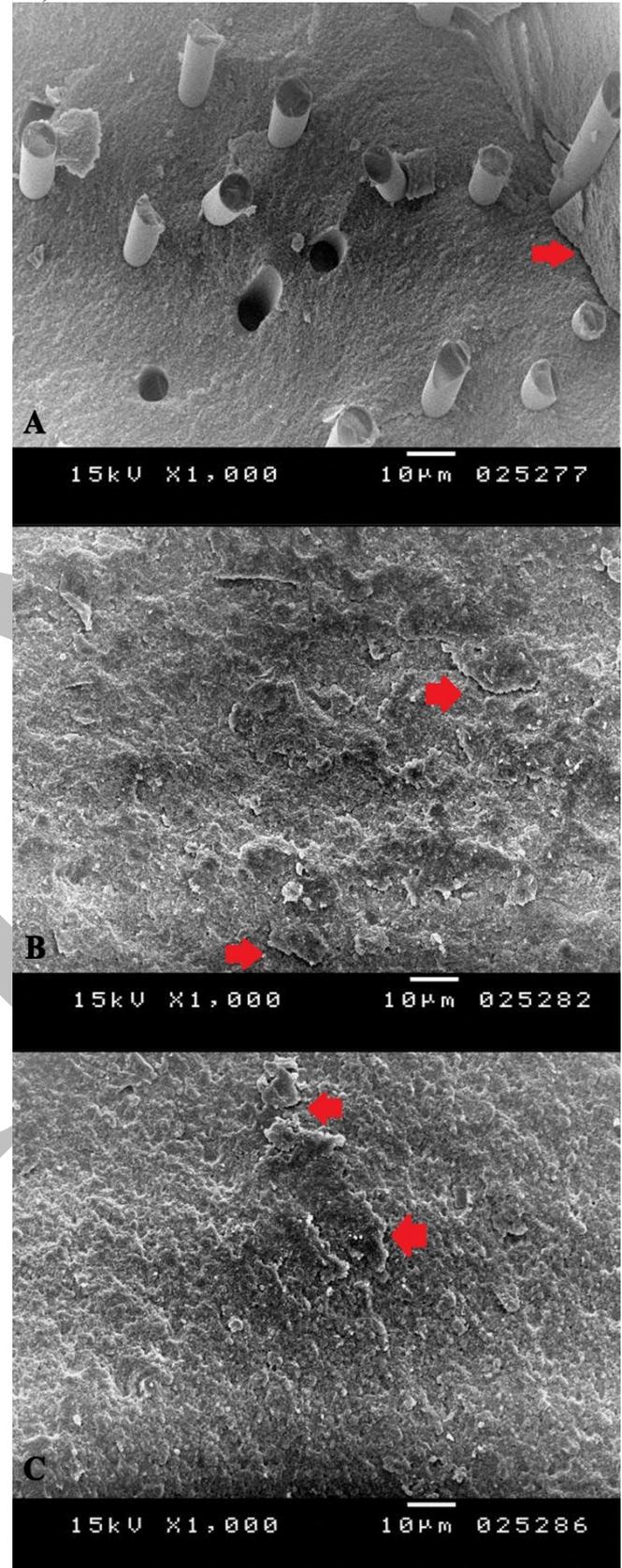


Figure 2: SEM images of flexural strength specimens after aging for 6 months. The red arrows show cohesive fractures in the resin matrix. (A) In the fiber-reinforced composite, the fracture goes around the glass fibers with the glass fibers remaining intact. (B) Nano-hybrid ceramic filled composite and (C) Nano-hybrid zirconia filled composite, show a clear cohesive fracture in the resin matrix and the ceramic fillers and zirconia fillers respectively.

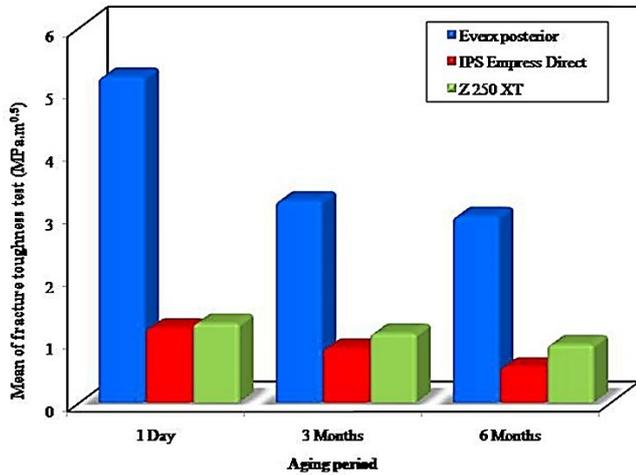


Figure 3: Comparison between the different composite materials in the same aging period and its effect on the fracture toughness.

Table 3: Fracture toughness results of the three composite materials at the three aging periods.

Fracture toughness test (MPa.m ^{1/2})	EverX Posterior (n= 7)	IPS Empress Direct (n= 7)	Z 250 XT (n= 7)	F test value
1 Day				
Mean ± SD.	5.21 ± 0.79	1.19 ± 0.04	1.27 ± 0.18	170.542*
Significance between groups	p ₁ <0.001*, p ₂ <0.001*, p ₃ = 0.752			
3 Months				
Mean ± SD.	3.23 ± 0.19	0.87 ± 0.16	1.11 ± 0.17	391.692*
Significance between groups	p ₁ <0.001*, p ₂ = 0.018*, p ₃ = 0.018*			
6 Months				
Mean ± SD.	2.98 ± 0.27	0.57 ± 0.12	0.93 ± 0.15	327.283*
Significance between groups	p ₁ <0.001*, p ₂ < 0.001*, p ₃ = 0.002*			
Significance between aging periods				
EverX Posterior	p ₄ <0.001*, p ₅ <0.001*, p ₆ = 0.349			43.101*
IPS Empress Direct	p ₄ <0.001*, p ₅ <0.001*, p ₆ <0.001*			50.044*
Z 250 XT	p ₄ = 0.086, p ₅ = 0.001*, p ₆ = 0.056			7.450*

p₁: p value for comparing between EverX Posterior and IPS Empress Direct
 p₂: p value for comparing between Everx Posterior and Z 250 XT
 p₃: p value for comparing between IPS Empress Direct and Z 250 XT
 p₄: p value for comparing between 1 day and 3 months
 p₅: p value for comparing between 1 day and 6 months
 p₆: p value for comparing between 3 months and 6 months
 *: Statistically significant at p ≤ 0.05

DISCUSSION

A- Flexural strength test

One of the laboratory tests most used to evaluate the mechanical behavior of resin composites is the flexural strength test. It simulates the complex forces that develop in areas of stress concentration, since it induces compressive and tensile forces simultaneously, close to and opposite from the point of loading, respectively (11). The test serves as a predictor of the mechanical behavior of a resin composite used clinically as stated by Reinhardt et al (1994) (20).

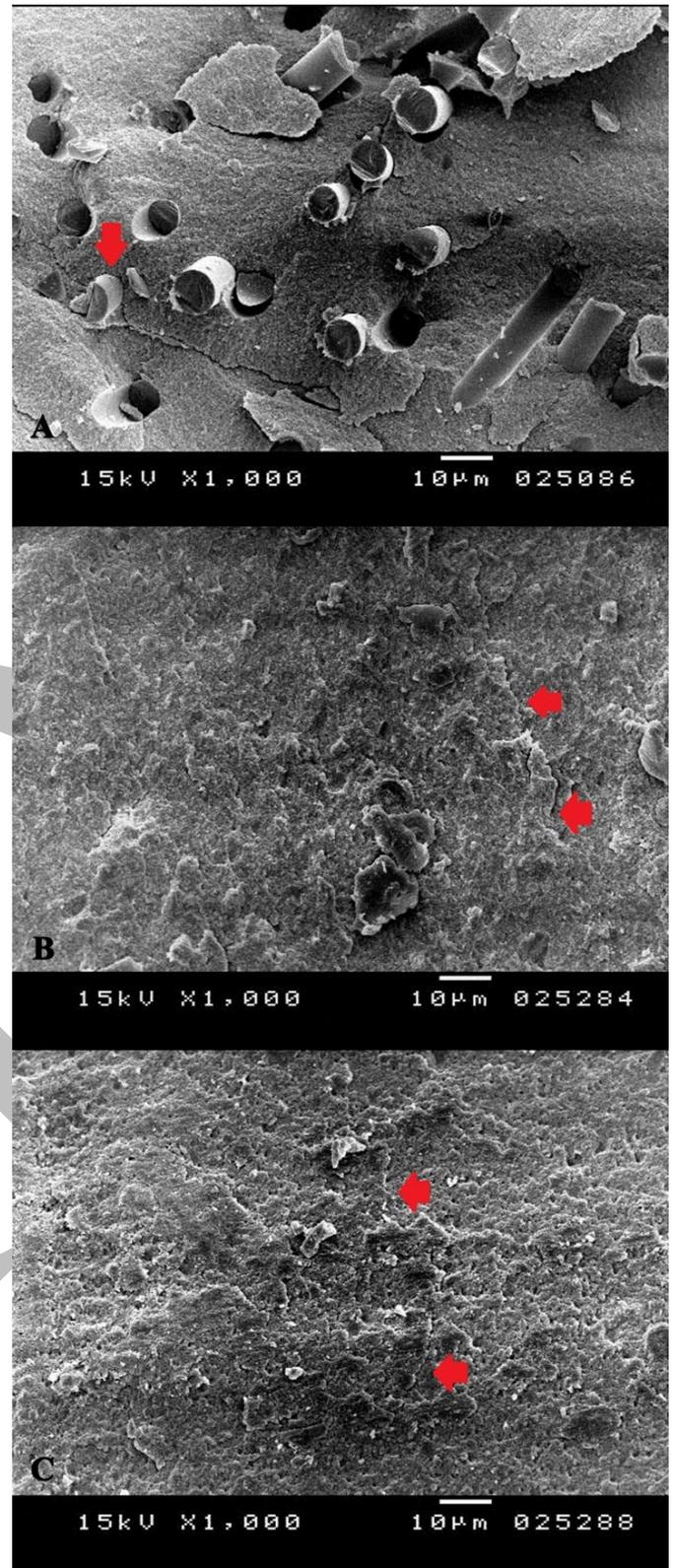


Figure 4: SEM images of fracture toughness specimens after aging for 6 months. The red arrows show micro-cracks in the resin matrix.(A)In the fiber-reinforced composite , the fibers acted as micro-crack deflectors .(B)Nano-hybrid ceramic filled composite and (C) Nano-hybrid zirconia filled composite, the ceramic fillers and zirconia fillers failed to act as micro-crack deflectors respectively.

Multiple factors influence the flexural strength test one of which is the test specimen dimensions. In this study specimens were prepared according to ISO standard 4049 with dimensions 25x2x2 mm (length x width x height)

which is the most commonly used specimen dimensions in literature (11).

Another factor that influences flexural strength test is the storage condition. Long-term water storage leads to reduced mechanical properties. This accelerates hydrolytic degradation of both the resin matrix and the silane layer covering the inorganic fillers (21).

The temperature is another important factor affecting flexural strength results. It has been argued that all mechanical properties must be evaluated at 37°C to bear clinical significance, since that is the temperature at which the materials will be used in the oral cavity (22).

Flexural strength results of this study showed that the fiber-reinforced composite had the highest flexural strength compared to the two nano-hybrid composites in all the three aging periods. This agrees with Abouelleil et al 2015 (23) who did a study to compare the mechanical properties of a newly developed fiber-reinforced composite (EverX Posterior, GC, Europe) to other commercially available bulk fill composites. They found that after 48 hours' storage in water, the fiber-reinforced composite (EverX Posterior, GC, Europe) had the highest flexural strength among the tested materials.

This also agrees with Goracci et al 2014 (24) who compared the flexural strength of the fiber-reinforced composite (EverX Posterior, GC, Europe) with other low-stress behavior dental composites. They found that the fiber-reinforced composite had the highest flexural strength among the tested materials after 24 hours' storage in water.

This is due to the fact that the fibers in the fiber-reinforced composite being longer (2 mm) than the critical fiber length of E-glass fibers (0.5-1.6 mm) with Bis-GMA polymer matrix, allows for stress transmission from matrix to fibers, thus producing an effective reinforcement. The physical explanation of the strengthening and stiffening mechanism is that since the matrix has a much lower modulus than the fiber, the matrix strains more. The critical fiber length is therefore the minimum length at which the center of the fiber reaches its ultimate tensile strength when the matrix reaches its maximum shear strength (25).

Additionally, the so-called Semi-Interpenetrating Polymer Network (Semi-IPN); a resin matrix containing Bis-GMA, TEGDMA, and PMMA in the form of net-poly (methyl methacrylate)-inter-net-poly (bis-glycidyl-A-dimethylacrylate) increases the mechanical properties (26).

Moreover, as shown in previous works, the filler volume percentage is closely related to the flexural strength and flexural modulus values. Interestingly, the fiber-reinforced composite with the lowest filler volume percentage (53.6%) had the highest flexural strength, showing the role of the fibers in increasing the material stiffness and resistance to bending force during testing and probably during function (23).

The results of this study showed that the flexural strength of the fiber-reinforced composite decreased significantly by about 17% after 3 months aging and 23% after 6 months aging. This may be due to plasticization of the resin matrix and loss of interfacial bond strength. A partial hydrolysis of the silane bonds formed between the glass fibers and the matrix may explain this result. Furthermore, the degradation of the glass fiber itself cannot be ruled out, because glasses also are susceptible to hydrolytic degradation. The mechanism of hydrolytic decomposition is based on the dilution of boric oxide, a compound which forms glass, from the surface of the glass fiber (27).

Hydrolysis is known to be an autocatalytic reaction, but it is possible that hydrolysis of the silane-promoted adhesion could also be reversible. This is due to silane coupling agent on the glass surface reforming the original bond by a recondensation reaction of the hydrolysis products, i.e., silanols to make a new bond with adjacent groups (27).

The primary mechanism for the ingress of water is diffusion and some absorption is facilitated by the polarity of polymer chains. Water molecules penetrate into the spaces between polymer chains and occupy positions between the chains, and thus, the polymer chains are forced apart. Water molecules act as a plasticizer and the polymer chains generally become more mobile and as a result, the flexural strength is reduced (27).

In this study, the nano-hybrid ceramic filled composite had the lowest flexural strength among the three tested composite materials in all the three aging periods. This may be due to the brittle nature of the ceramic fillers used (Ytterbium trifluoride) which can't withstand bending and break immediately without permanent deformation.

In addition to that, the resin matrix of the nano-hybrid ceramic filled composite is made of Bis-GMA and UDMA. Bis-GMA, despite its high intrinsic reactivity, the presence of hydroxyl groups on the backbone and the pi-pi bond interactions given by the aromatic rings increase the initial viscosity to a point that the polymer typically does not reach high conversion which leads to increased water sorption and consequently low flexural strength values (28).

UDMA is also characterized by high solubility, hydrophilicity and low degree of conversion (28) which may explain why the flexural strength of IPS Empress Direct was lower than the other two composites. In the literature it is also reported that UDMA based composites undergo softening in water or oral simulating fluids easier than Bis-GMA resins (29).

The nano-hybrid zirconia filled composite had the second highest flexural strength after the fiber-reinforced composite (EverX Posterior, GC, Europe). The fillers in the nano-hybrid zirconia filled composite are surface-modified zirconia/silica with filler volume (68%) which is higher than that of nano-hybrid ceramic filled composite (52-59%) and the resin matrix is a blend of 5 polymers; Bis-GMA, UDMA, Bis-EMA, TEGDMA and PEGDMA. This explains its superiority over (IPS Empress Direct, Ivoclar Vivadent, Lieschtenstein) but still it was inferior to the fiber-reinforced composite (EverX posterior, GC, Europe) with its fibers.

The flexural strength results are consistent with the scanning electron microscope (SEM) imaging findings. SEM images revealed cohesive fractures in the resin matrix. The fibers remained intact in the fiber-reinforced composite while in the two nano-hybrid composites, the fracture line passed through the matrix and fillers. These findings emphasize the role of fibers in increasing the flexural strength of dental composites.

B- Fracture toughness test

Dental materials researchers regard the fracture toughness of a material as a more accurate predictor than traditional compressive and tensile testing of how a material will perform under various occlusal & masticatory stresses (12).

Fracture toughness (K_{IC}) is a measure of the stress intensity at the tip of a crack or flaw from which a crack propagates through a material in an unstable manner. The subscript (I) refers to the case when the crack is propagated in mode I or tensile opening. This property has been related

to the ability of the material to resist both crack propagation and wear in the oral environment (19).

The pre-crack is required in fracture toughness testing, because it simulates a sharp, natural flaw in the interior of a material. Since the stress concentration is highest when the notch or crack is sharpest, it was believed that the most accurate evaluation of fracture toughness for dental composites would be achieved by testing specimens with an extremely sharp flaw, i.e., one made by propagating a crack from a sharp notch. The most commonly used fracture toughness test in literature is the single-edge notch test with precracks and has recorded the most accurate and predictable fracture toughness values (30).

Results of this study showed that the fiber-reinforced composite had the highest fracture toughness compared to the two nano-hybrid composites in all the three aging periods. The results are in agreement with Abouelleil et al 2015 (23) and Goracci et al 2014 (24).

Reinforcing effect of the fiber fillers is based on stress transfer from polymer matrix to fibers. Random fiber orientation and the polymer matrix by the semi-IPN structure likely had a significant role in mechanical properties. In addition to the toughening mechanism by fibers, the linear polymer chains of PMMA in the cross-linked matrix of BisGMA-TEGDMA plasticize the polymer matrix to some extent and increase the fracture toughness of the composite resin (26).

In this study, the nano-hybrid ceramic-filled composite showed the least fracture toughness at all the three aging periods. This is attributed to the ceramic fillers and their brittle nature and their failure to act as crack stoppers. Also the hydrophilicity of its UDMA based resin matrix reduced its fracture toughness.

The majority of studies are in general agreement that the fracture toughness of composites increases as filler volume fraction is increase The presence of reinforcing particles distributes the propagating nominal force into many components, causes the crack front to curve or dissipate between particles, and becomes energetically unfavorable for a crack to grow (31).

The results of the study showed that the nano-hybrid zirconia filled composite with filler volume (68%) had higher fracture toughness than the nano-hybrid ceramic filled composite with filler volume (59%). But although the fiber-reinforced composite had the lowest filler volume (53.6%), it showed the highest fracture toughness. This shows the ability of fibers to act as crack deflectors rather than ceramic or zirconia fillers.

Besides the matrix-filler interaction, crack pinning, crack branching, crack deflection, or micro crack-induced toughening are seen as main mechanisms of increasing fracture toughness values by filler particles in resin composites. The mechanism of increasing toughness by matrix-filler interaction seems to play an important function especially in composites with decreased filler size, like the nano-hybrid materials. The decreased filler size is able to change the organic matrix between the particles, improving the mechanical properties, as a consequence of decreasing inter-particle distances (31).

The fracture toughness results are consistent with the scanning electron microscope (SEM) imaging findings. SEM images revealed that the fibers acted as crack deflectors in the fiber-reinforced composite while in the two nano-hybrid composites, the ceramic and zirconia fillers

failed to act as crack deflectors. These findings emphasize the role of fibers in increasing the fracture toughness of dental composites.

CONCLUSIONS

From the results of this study, the following could be concluded:

The fiber-reinforced composite (EverX posterior,GC,Europe) had the highest flexural strength and fracture toughness compared to the two nano-hybrid composites (IPS Empress Direct, Ivoclar Vivadent, Lieschtenstein) and (Z250 XT,3M ESPE ,USA) in all the three aging periods. Although aging in water decreased the mechanical properties of the fiber-reinforced composite, but it still remained higher than the two nano-hybrid composites which ensures its ability to withstand forces of mastication in the posterior region.

CONFLICT OF INTERESTS

The authors declare that they have no conflict of interests.

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