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EVALUATION OF FRACTURE TOUGHNESS AND MICRO HARDNESS OF THREE CURRENT RESIN COMPOSITE RESTORATIVE MATERIALS

Hala Mohammad Fares*

ABSTRACT

Various different resin composite restorative materials are continuously being introduced to the market. Their composition and mechanical properties, largely, influence their clinical performance.

Materials and methods : Three resin composite restorative materials (Tetric N – Ceram, ACTIVA, and the hybrid resin composite Te - Econom Plus) were investigated regarding fracture toughness and micro-hardness. Thirty disk shaped samples (n = 10 / group), were prepared, light cured and finished and polished. The surface micro-hardness was assessed using a Digital Display Vickers Micro-hardness Tester. Both fracture toughness and micro-hardness were assessed using the indentation technique with heavy loading.

Results : Regarding both fracture toughness and micro-hardness, the highest means plus or minus SD were recorded by Tetric N – Ceram, followed by Te – Econom Plus, while ACTIVA demonstrated the least.

KEY WORDS : Fracture toughness, micro hardness, resin composites.

INTRODUCTION

Resin composites are vastly and increasingly used in dental restorative procedures due to their appropriate mechanical, esthetic, and physical properties as well as improvements in adhesive dentistry [1–6]. The recent developments in fillers types and content as well as in polymers, have lead to a broad spectrum of resin composite materials that fulfil the requirements of each clinical situation[2,7]. Resin composite materials should demonstrate appropriate mechanical strength to endure forces in high stress- bearing areas, or else these forces could induce bulk fracture and destruction of restorations[1]. Therefore, resin composite materials

* Associate Professor, Conservative Dentistry Faculty of Dentistry Pharoahs University Cairo Nasr City

used for anterior teeth restorations such as in class III or class IV restorations at the incisal edges; should be entirely fracture- resistant to withstand the high flexural forces [8]. Moreover, the resin composite materials behavior against complex stresses including shear, compressive and tensile stresses, should be assessed[2,7]. Fracture toughness has been defined as a materials resistance to crack propagation[3,9]. It is one of the most important and outstanding mechanical properties to be studied as it reveals the materials behavior with different clinical situations [3]. Moreover, fracture has been reported to be at the top of the most frequent failures of dental restorations as regards to dental practice[3,10]. The surface micro-hardness of a material reveals how hard the material is to resist indentation. The higher the micro-hardness of a material the higher its expected mechanical properties [3,11–13]. Generally, micro-hardness is a measure of the resistance of a material to plastic deformation and wear [14]. The different mechanical properties and chemical compositions of resin composites are among various factors that influence their clinical performance [15]. In this study, an evaluation of the fracture toughness and micro-hardness of three recent resin composite restorative materials [Tetric N – Ceram, ACTIVA, and the hybrid resin composite Te – Econom Plus] was carried out.

MATERIAL AND METHODS

Three direct resin composite restorative materials were assessed for micro-hardness (VHN) and fracture toughness properties. The materials included the nano hybrid resin composite restorative material Tetric N-Ceram, ACTIVA and the hybrid composite restorative material (Te-Econom Plus). The composition of each tested material was presented in Table 1.

TABLE (1) Composition and manufacturers of the resin composite materials used in this study (data provided by the manufacturers)

Resin Composite	Composition	Manufacturer
Tetric N-Ceram shade (A2) (Nanohybrid resin composite restorative material	Resin matrix; DMA: 19–21% by weight, plus ethyoxylated bisphenol A DMA, Bis-GMA (3–<10%) Filler;(more than 81% by weight) Barium glass, ytterbium, trifluoride, in addition to mixed oxides	IvoclarVivadent, Schaan, Liechtenstein Lot# Z01G6B
ACTIVA BioACTIVE Restorative shade (A2)	Resin matrix; Blend of di Urethane and other methacrylates with a modified polyacrylic acid (44.6%), contains no bisphenol A, no Bis-GMA, no BPA derivatives Filler; that in addition toAmorphous silica (6.7%), and sodium fluoride(0.75%)	Pulpdent Corp, MA, USA Lot# 140611
Te-Econom Plus (Hybrid Composite Restorative material) shade (A2)	Resin matrix; Bis-GMA, Urethane dimethacrylate (UDMA), with triethylene glycol dimethacrylate (TEGDMA) (18.8% by weight) in addition to a small amount of catalyst, stabilizers and pigments (0.21% by weight). The inorganic filler comprises (81% by weight. The filler size is (0.7ìm).	IvoclarVivadent, UK Lot# Z01W5S

Bis-GMA: bisphenol A, diglycidyl ether methacrylate, TEGDMA: triethylene glycol dimethacrylate; UDMA: urethane dimethacrylate

The composite resin materials were used for preparing the 30 samples (n=10/group) of 10 mm diameter and 2 mm thickness. A circular Teflon mold 10 mm in diameter and 2.0 mm in thickness was used. On a glass slide, the teflon mold was situated, then filled with light curing resin composite and covered with a matrix strip. Another glass slide was placed on top of the matrix strip and gentle pressure was exerted to obtain a smooth surface. The materials were then light cured through the transparent matrix according to the manufactures' instructions, for 40 seconds; using an LED curing unit (Elipar S10 free light, 3M, ESPE, USA) with a spectral range of 450-470 nm wavelength and 1200mW/cm2 intensity on both top and bottom surfaces . Afterwards, the resin composite material was extruded from the mold by applying positive pressure using a pestle of 9.5 mm diameter in order to perform equal distribution of pressure. All the samples were stored in distilled water for 24h at 37^0C to ensure complete polymerization. Samples were finished and polished with 3M Sof-Lex disks to obtain a clinical finish.

Vickers Micro-hardness Test (VHN)

The surface micro-hardness of the samples was assessed by the aid of a Digital Display Vickers Micro-hardness Tester (Model HVS-50, LaizhouHuayin Testing Instrument Co., Ltd. China) using a Vickers diamond indenter supplied with a 20X objective lens. A load of 200g was exerted onto the surface of each specimen for 15 seconds. Three indentations, were made on the surface of each specimen. It was made sure that those indentations were equally spaced over a circle and were not closer than 0.5 mm to each other (figure 1). The lengths of the formed diagonals of indentations were measured by a built in scaled microscope as well as the Vickers . The obtained measurements were converted into micro-hardness values as follows : Micro-hardness calculations were performed, by utilizing the following equation: $HV= 1.854\ P/d^2$; where, HV is Vickers hardness in Kgf/mm2, P is the load in Kgf and d is the length of the diagonals in mm.

Fig. (1) Indentation mark left by the Vickers diamond indenter.

Fracture toughness assessment

In general, both hardness and fracture toughness measurements were assessed using the indentation technique[16]. The concept of the indentation technique lies in the fact that a heavy loading results in a series of cracks in a brittle material; around the Vickers diamond indenter. These cracks; when viewed from a top perspective; appear to originate from all the corners of the indentation (figure 2). The lengths of these cracks, as indicated by the surface dimension "c,"; increase with the elevation of the indentation load and is expressed as an inverse function of fracture toughness. The cracks lengths were measured using a built in scaled microscope. The fracture toughness was measured using the following equation:

$$K_{IC} = 0.016(E/H)\ 0.5\ (P/c^{1.5});$$

where K_{IC} is the fracture toughness, H is the Vickers hardness, E is the elastic modulus, P is the exerted indenter load, and c is the crack length; measured from the indentation center [17].

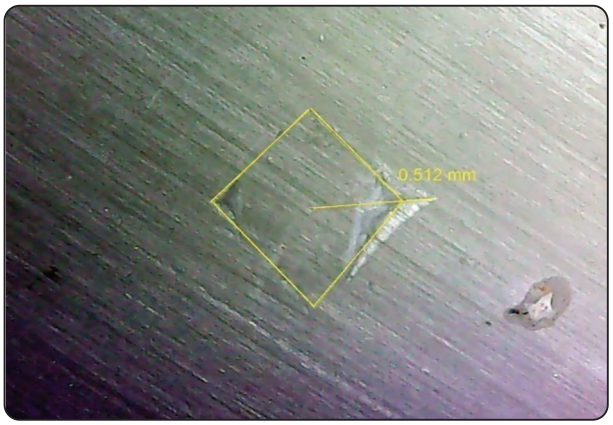

Fig. (2) Crack length (measured from the center of the indentation)

The data were presented as means, standard deviation (SD) values and 95% confidence intervals (low –high). All data were checked for normality by assessing the data distribution and using Kolmogorov-Smirnov and Shapiro-Wilk tests. After homogeneity of variance and normal distribution of errors had been confirmed, one-way analysis of variance was performed followed by Tukey's post-hoc test if significance was indicated. Sample size (n=10/group) was considered large enough to detect large effect sizes for main effects and pair-wise comparisons, with a satisfactory level of power set at 80% and a 95% confidence level.The results were analyzed by the aid of a Graph Pad Instat (Graph Pad, Inc.) software for windows. A value of P ≤ 0.05 was considered statistically significant.

RESULTS

Vickers micro-hardness(VHN)

Vickers micro-hardness (VHN) results (Means ± SD) for all groups are summarized in table (2) and figure (3).

It was found that the highest mean ± SD values of Vickers hardness were recorded for the ***Tetric N-Ceram*** group (77.815 ±2.231Kgf/mm^2); followed by the ***Te-Econom Plus*** group (means ± SD values 72.386 ± 3.231Kgf/mm^2); meanwhile the lowest mean ±SD values were recorded with ***ACTIVA*** group (59.980 ± 2.468Kgf/mm^2). The differences among groups was statistically ***significant*** as verified by the ANOVA test followed by the Tukey's post-hoc pair-wise tests (P=<0.0001<0.05) as shown in table (2) and figure (3).

TABLE (2) Vickers micro-hardness (Kgf/mm^2) results (Means ± SD) for all composite groups

Variable		*Descriptive statistics*		*ANOVA test*
		Mean±SD	*95% CI (low-high)*	*P value*
Composite group	*Tetric N-Ceram*	77.815^A±2.231	76.432- 79.198	<0.0001*
	ACTIVA	59.980^C± 2.468	58.451 – 61.509	
	Te-Econom Plus	72.386^B± 3.231	70.384 – 74.388	

Different subscript letter in the same row indicating statistically significant difference betweensubgroups (p< 0.05) CI; confidence intervals *; significant (p< 0.05) ns; non-significant (p>0.05)

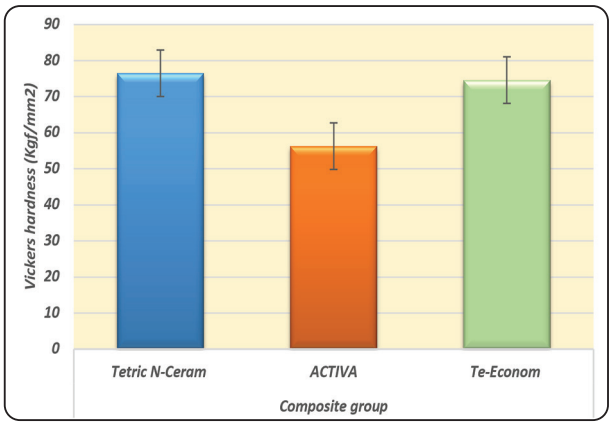

Fig. (3) Column chartcomparing the mean values of Vickers micro-hardnessbetween all groups

Fracture toughness (MPa.m$^{1/2}$)

Fracture toughness (MPa.m$^{1/2}$) results (Means ± SD) for all groups are summarized in table (3) and figure (4).

It was found that the highest means ± SD values of fracture toughness were recorded for the ***Tetric N-Ceram*** group (1.7385±0.0846 MPa.m$^{1/2}$); followed by the ***Te-Econom Plus*** group (means ± SD values 1.5877±0.0657MPa.m$^{1/2}$); meanwhile the lowest means ± SD values were recorded for the ***ACTIVA*** group (1.2073±0.0247MPa.m$^{1/2}$). The differences among groups was statistically ***significant*** as verified by the ANOVA test followed by Tukey's post-hoc pair-wise tests (P=<0.0001<0.05) as shown in table (3) and figure (4).

TABLE (3) Fracture toughness (MPa.m$^{1/2}$) results (Means ± SD) for all composite groups

Variable		*Descriptive statistics*		*ANOVA test*
		Mean ± SD	*95% CI (low-high)*	*P value*
Composite group	*Tetric N-Ceram*	1.7385^{A}± 0.0846	1.6860 - 1.7909	<0.0001*
	ACTIVA	1.2073^{C}± 0.0247	1.1919 - 1.2226	
	Te-Econom Plus	1.5877^{B}± 0.0657	1.5469 - 1.6284	

Different subscript letter in the same row indicating statistically significant difference betweensubgroups (p< 0.05)
CI; confidence intervals *; significant (p< 0.05) ns; non-significant (p>0.05)

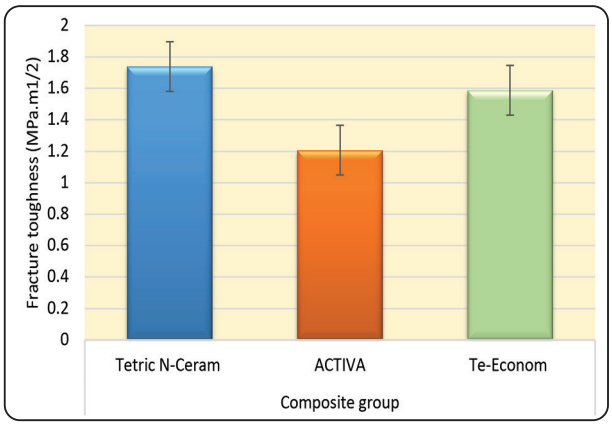

Fig. (4) Column chartcomparing the mean values of fracture toughness between all groups

DISCUSSION

The results of this study revealed that, in regard**,** for both fracture toughness and surface micro-hardness, the highest means plus or minus the standard deviations were demonstrated by the nano hybrid resin composite restorative material (Tetric N–Ceram), followed by the hybrid resin composite (Te–Econom Plus); meanwhile the ACTIVA resin composite showed the least means. In general, the mechanical and physical properties of resin composite restorative materials, are, largely, dependent on the fillers content, type, size, loading level, and morphology; as well as the matrix content

and chemical composition. The two hybrid resin composites (the nano hybrid Tetric N – Ceram, and the hybrid Te–Econom Plus); possess adequate fillers content (about 81 % by weight). Increasing the fillers content has been reported to result in higher hardness and fracture toughness [1,18–20]. Also, the fillers particle size, and loading level have a great impact on the fracture toughness and surface micro-hardness [1,2,7]. Both hybrid resin composites (the nano hybrid Tetric N–Ceram, and the hybrid Te – Econom Plus); have adequate fillers particle size and loading levels which accounts for their, relatively, high surface micro-hardness and fracture toughness. However, the nano hybrid resin composite (Tetric N–Ceram); demonstrated, significantly, higher surface micro-hardness and fracture toughness. This could be attributed to the nano-sized filler particles which tend to get inserted in the nano spaces between the other filler particles; thus producing a very high filler loading level. Also, the spherical-shaped symmetric nano fillers contribute to the superior mechanical and physical properties[21,22]. The ACTIVA resin composite demonstrated the least values of fracture toughness and surface micro-hardness; which could be attributed to the, largely, reduced fillers content. Regarding, the organic matrix, the ACTIVA resin composite contains a large amount of organic matrix (44% by weight); which accounts for the reduced mechanical properties including the surface micro-hardness and fracture toughness. Also, the chemical composition of the organic matrix has an influence on the mechanical and physical properties of resin composite restorative materials. The matrix structure of both hybrid resin composites (the nano hybrid resin composite Tetric N–Ceram, and the hybrid resin composite Te–Econom Plus); is combined with UDMA and TEGDMA monomers which could have been the reason for the rising amounts of polymer cross links[23,24]. The rapid turnover in the production of new esthetic restorative materials demands that researches be, continuously, evaluating the mechanical and physical properties of these materials.

CONCLUSION

The mechanical and physical properties of resin composite restorative materials are, greatly, influenced by the fillers content, type, size, loading level and morphology; in addition to the matrix content and chemical composition.

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