Original Article

Evaluation of Mechanical Properties of Newer Nanoposterior Restorative Resin Composites: An *In vitro* Study

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Abstract

Introduction: Tooth coloured restorative materials are subjected to various physical, mechanical conditions in oral conditions. Many newer composites with improved physical and mechanical properties are introduced for clinical use. There are not many clinical studies on recent composites. The aim of this study was to compare the mechanical properties of five commercially available nano composite restorative materials. **Materials and Methods:** Specimens of five nano posterior composite SureFil SDR, ClearFil Majesty, Ever X, Tetric Evo Ceram bulk fill and Filtek Z350 were tested in the study. All samples were prepared According ISO 4049 and polymerized with a LED light for 40 seconds and subjected to mechanical tests for compressive strength, flexural strength, flexural modulous and nano hardness. **Statistical Analysis:** Results obtained were subjected to one way ANOVA and Turkey's post hoc test at significance (p <0.05). **Results:** There was significant differences among composites restorative resins tested. CFM Nano hybrid composite exhibited highest hardness values. Flexural strength, flexural modulous and hardness properties of Ever X and Z350 were almost similar. Compressive strength value of Ever X was high compared with other four composites. SDR exhibited least values when compared with other composites. **Conclusion:** Differences in compressive strength, hardness, flexural strength and modulous is due to differences in percentage and type of filler particles in all composite resin material tested.

Keywords: *Mechanical properties, nanohardness, nanoindentation test, posterior composites*

Introduction

Tooth-colored restorative resins are the most preferred restorations due to improvements in their physical, mechanical, and optical properties and ease in clinical handling. Different fillers and monomer systems are modified or added to restorative materials recently for the success of restorations clinically. Restorative resins are modified from past to present from macrofilled composites, microfilled composites, hybrid composites, microhybrid composites, and flowable composites to recent bulk fill composites and nanocomposites. Improvements are mainly aimed at reducing polymerization shrinkage and increasing hardness, compressive strength, flexural strength, and flexural modulus by introduction of newer resin formulations and filler concentration.[1,2] Improvements in higher modulus of elasticity, greater flexural strength, compressive strength, diametrical tensile strengths, hardness, fracture toughness, and wear resistance of these newer composite resins have been reported in previous studies.[3] Nanocomposites thereby respond much better to the functional stresses of mastication as compared to the conventional resins. Restorative materials used in stress-bearing areas have to be tested for physical and mechanical properties such as high strength, fracture toughness, surface hardness, optimized modulus of elasticity, low wear, low water sorption and solubility, low polymerization shrinkage, low fatigue and degradation, high radiopacity, and optical properties as these are still the major concern of composite materials' success clinically. Nanohybrid composites contain the least amount of organic matrix and greater percentage of fillers and demonstrate lesser polymerization shrinkage than the nanofill composites.[4,5] Recent composite materials available have variation in composition and viscosity, and hence need to be tested for all parameters.

Hardness is described as resistance to indentation. Hardness test evaluates the

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Table 1: Composition of composite restorative resin materials

Group/trade name	Composition	Characteristic	Shade	Lot no
A-Surefil SDR (Dentsply)	UDMA, di methacrylate resin, di functional diluents, barium and strontium alumina -fluoro-silicate glasses, 68%wt and 45% vol, photo initiators and colouring agents	Flowable composite (bulkfill)	Universal shade	11221
B-Clearfil majesty posterior (KurarayEurope)	BisGMA, TEGDMA, hydrophobicaromatic, dimethacrylate, camphorquinone, accelerators, pigment, others Fillers: silanated glass ceramic fillers1.5µm, surface treated alumina micro fillers 20 nm.Filler load 92% wt, (82%vol)	Nanohybrid	A2	00122C
C-EverXP, (EXP, GC Europe)	Bis-GMA10-20%, TEGDMA-5-10%, Silicondioxide 5-10%, barium glass 60-70%, glass fiber 5-15%, polymethylmethacrylate, photoinitiators	Fiber reinforced (bulkfill)	Universal shade	1307022
D-TetricEvo Ceram, Ivoclar, vivadent	Bis-MA, UDMA 19.7%wtBarium glass, ytterbium tri fluoride62.5%wt, mixed oxide and prepolymers19.7%wt	Nano hybrid (bulkfill)	A2	S08629
E-Filtek Z350 3M (ESPE)	Bis -GMA, UDMA, TEGDMA, Bis EMA, discrete nonagglomerated and nonaggregated silica and zirconia fillers of 20 nm and 4-11 nm in size.	Nano composite	A2	N562394

Bis EMA: Ethoxy bisphenol A diglycidyl dimethaacrylate; Bis-GMA: Bisphenol ether dimethacrylate; TEGDMA: Triethylene glycol dimethaacrylate; UDMA: Urethane dimethacrylate

degree of polymerization of resin composites. Hardness and depth of cure of these materials can be obtained using Vickers and Knoop microhardness test.[6] Nanoindentation test is one of the methods used to measure the mechanical properties (hardness) of materials. The principle uses the same technique as microindentation, but with much smaller probe and loads so as to produce indentations from less than a hundred nanometers to a few micrometers in size.[7] Compressive test is applied to compare dental amalgam, composites, impression materials, investments, and cement to be used in stress-bearing areas clinically.[8] The flexural strength of a material is its ability to bend before it breaks or the fracture resistance of a material.[9] It is obtained when the ultimate flexibility of one material is achieved before its proportional limit.[3] Flexural forces are the result of forces generated in clinical situations, and the dental materials need to withstand repeated flexing, bending, and twisting. A high flexural strength is desired once these materials are under the action of chewing stress that might induce permanent deformation.[9] The aim of the present study was to investigate the mechanical properties of five different commercially available dental composites having different organic matrix, filler loading, and filler types, under the same curing and testing conditions. The null hypothesis was that there is no significant difference in mechanical properties (flexural strength, flexural modulus, compressive strength, and nanohardness) among the newer posterior nanocomposites.

Materials and Methods

Five commercially available posterior composites, namely SureFil SDR Posterior composite (DENTSPLY), ClearFil Majesty Posterior Composite (KURARY), EverX Posterior (EXP,GC Europe), Tetric Evo Ceram bulk fill Posterior Composite (VIVA DENT), and Filtek Z350 (3M), were tested in the study and divided into five groups and named as A, B, C, D, and E, respectively. Their composition is depicted in Table 1.

Preparation of samples

All samples were prepared according to ISO 4049.

Nanohardness test

Ten specimens for each group were prepared in cylindrical plastic molds (8 mm diameter × 2 mm depth) which were placed on a glass microscope slide, filled with material and covered with a polyester strip and a glass slide, taking care to obtain a flat surface without any defects and entrapped air. The materials were polymerized by light-emitting diode (LED) light-curing unit LEDition (Ivoclar Vivadent AG, schaan, Liechtenstein) with 40-s exposure to each specimen's top and bottom surfaces. The samples were stored in individual light-protected plastic tubes with distilled water at 37°C for 24 h. After this step, the samples were tested in nanohardness equipment Nanomechanical test instrument, The TI 950 TriboIndenter, and Hysitron TI 950 (St. Minneapolis, USA) with force range available for nanomechanical testing (≤30 nN to 10 N).

Flexural strength test

Six samples of each composite resin were made using a 25 mm × 2 mm × 2 mm (length, width, and height) glass mold. Flexural strength and flexural modulus were determined in a three-point-bending test, using a universal testing machine. Bar-shaped specimens, measuring 2 mm × 2 mm × 25 mm, were produced by applying the composites to a stainless Steel mold and were then shaped between two parallel glass plates, covered with transparent matrix strips prior to light curing. Irradiation done on the top and bottom of the specimens, in order to prevent multiple polymerizations. After removal from the mold, the specimens were finished to smooth surface in order to get rid of disturbing edges or bulges. Thirty such specimens of all groups were produced and then stored in distilled water for 24 h at 37°C, 20 of them being loaded to fracture right after the 24-h water storage.

Table 2: Mean compressive, flexural strength and hardness values of the materials tested

Group	Compressive Strength (MPa)		Flexural Strength (MPa)		Flexure Modulus (MPa)		Nano Hardness (GPa)	
	Mean (MPa)	Std. Dev	Mean (MPa)	Std. Dev	Mean (MPa)	Std. Dev	Mean (MPa)	Std. Dev
A-Surefil SDR	349.08	59.89	71.20	7.37	24.07	2.50	1331.64	79.29
B-Clearfil majesty posterior	341.70	90.31	58.51	16.19	17.17	4.79	866.16	60.44
C-EverX, GC Corporation, Japan	409.58	63.00	92.40	28.54	33.75	4.07	1022.27	120.22
D-TetricEvo Ceram, Ivoclar, vivadent	258.12	72.91	70.16	0.17	23.22	0.24	1232.25	98.98
E-Filtek Z350 3M (ESPE)	226.48	68.80	97.58	21.40	32.53	7.14	1049.31	48.07

Compressive strength test

Six samples of each composite resin were made using a polytetrafluoroethylene mold (3 mm in diameter and 6 mm in height). The samples were placed in a universal testing machine at a crosshead speed of 1.00 mm/min.[9,11] Data were obtained in kgf and transformed into MPa using the following formula: $RC = F \times 9.80/A$, where RC is the compressive strength (MPa), F is the recorded force (kgf) multiplied by the constant 9.80 (gravity), and A is the base area (7.06 mm²).

Statistical analysis

Results were subjected to one-way ANOVA for comparison between groups and Tukey's *post hoc* test to compare the composites among groups. Statistical analysis was performed using software (SPSS version 20.0 Inc., Illinois, USA) at significance ($P < 0.05$).

Results

Table 2 shows the nanohardness indentation values (MPa), of which Z350 showed the highest nanohardness values (indication depth of indentation). However, there was no significant difference in the mean nanohardness values of CFM™ and SDR™. The nanohardness values of Z350 were better followed by CFM™ > SDR™ > TEC™ > EX™. The greater the depth of penetration, the less is the hardness values. Table 2 shows the compressive strength (MPa), of which CFM™ showed the highest compressive strength. However, there was no significant difference in the mean compressive strength of TEC™ and Z350. There was a significant difference between Ever X and the other composite resins. The compressive strength of CFM was better when compared to that of TEC™ (> Z350 > EX™ > SDR™). Table 2 shows the flexural strength (MPa), of which Filtek™ Z350 showed the highest flexural strength followed by EverX™ > SDR™ > TEC™ > CFM™. There was a significant difference between Z350 and the other composites. Table 2 shows the flexural modulus (GPA), of which Ever X™ showed the highest flexural modulus followed by Z350 > SDR™ > CFM™ > TEC™. There was a significant difference between EverX™, Z350, and the other composites.

Discussion

Among all the restorative materials available, composite resins are becoming the material of choice for restoration of all teeth. In recent times, a variety of dental resin composite restorative materials are available in the market for clinical use. Manufactures claim the materials to be superior in providing good esthetics, color stability, mechanical properties, compatibility with oral tissues, and longevity of restorations. The present composite resins marketed are aimed at providing better esthetic and optical properties, wear resistance, easy handling, and reduced polymerization shrinkage because of changes in the composition of resin matrix, filler type, percentage of filler loading, and particle size. In the present study, comparing the results obtained, the null hypothesis was rejected as there was significant difference in mechanical properties (flexural strength, flexural modulus, compressive strength, and nanohardness) among the newer posterior nanocomposites tested.

The restorative materials used in oral environment are subjected to various occlusal forces.

In the present study, the materials were investigated for compressive strength, flexural properties, and surface hardness for long-term performance of materials in oral conditions. There were significant differences between groups. CFM™ showed the highest compressive strength values followed by TEC™, Z350, and EX™ and the least values with SF™. EverX™ showed the highest flexural strength followed by Z350, CFM™, and SDR™, and TEC™ and EverX™ showed the highest flexural modulus followed by CFM™, EX™, SDR™, and TEC™. Filtek™ Z350 showed superior hardness value followed by CFM™, SDR™, TEC™, and EX™. These results are in agreement with a previous study.[11]

In this study, flexural strength and flexural modulus were tested. This test indicates the material resistance to fracture to masticatory forces; in the present study, EverX™ showed the highest and TEC™ showed the lowest values. Abouelleil *et al*., Ilie *et al*., Garoushi *et al*., and Czasch and Ilie showed similar results in using bulk fill composites in their studies, concluding that filler volume percentage is closely related to the flexural strength and flexural modulus values.[10-13] The total inorganic fiber and filler content of EverX™ posterior is 77% by weight and 53.6% by volume. the average size of the radio-opaque barium-silicate filler particles is between 0.1 and 2.2 μm and the average length of an individual glass fiber is between 1 and 2 mm; the particles performed better in both the tests compared to other groups. Results obtained are in accordance with previous studies.[11]

Similar improved mechanical properties of EverX™ posterior were reported by Garoushi *et al*.[12] in their study. The reason for fracture resistance could be because of glass fibers which increase the material stiffness and resistance to bending forces, which is very important for a restorative material to function in oral conditions.[11] CFM™ showed less values, the reason being the composite is nanohybrid and contains 82% filler by volume, making it less flexible compared to EX™, Z350, SDR™, and TEC™. Kim *et al*. in their studies concluded that composites with the highest filler by volume exhibited the highest values of flexural strength, flexural modulus, hardness, and fracture toughness.[14]

Compressive strength test evaluates the masticatory forces of restorative material especially posterior composites. In the present study CFM showed highest compressive strength values when compared to TEC, Z350, EXP and SDR. CFM is a nano hybrid composite contains 82% filler volume , according to Kim *et al*.[15] the larger size of filler particle and high percentage of fillers reduces the crack formation and deflection in composites making the material resistant to fracture, this might be the reason for better compressive strength results with CFM. The TEC is a nano hybrid composite consisting of monomer Bis-GMA, UDMA and Bis -EMA and different type of isofillers 61% filler volume. These isofillers are specially designed to reduce polymerisation shrinkage and improve other mechanical properties. The reason for better compressive strength could be because of decrease in inter-particle distance between the nanofiller which reduces the tendency for crack formation and propagation and the smooth and rounded edges of the spherical nanoparticles tends to distribute stress more evenly throughout the composite resin which is almost similar to study concluded by De Moraes *et al*.[16] TEC showed better results compared to Z350 containing 58.4% filler volume, EXP containing 53.6% filler volume and SDR 45 % filler volume. EXP has glass fibers as filler particles showed almost similar results as TEC. SDR has 45% filler volume showed the least values shows the role of importance of fillers in composites to be used in stress bearing areas.[17]

The nanohardness test showed the highest Values with CFM™ followed by EX™, Z350, TEC™, and SDR™. The results obtained in this study clearly indicate the relation between type and percentage of fillers in the hardness of composite materials. CFM™ is a nanofilled posterior composite composed of resin matrix Bis-GMA, TEGDMA, hydrophobic aromatic, dimethacrylate, and nano- and microinorganic filler [Table 1], with a surface coating incorporating more fillers in resin matrix with 92% weight and 82% volume, which improves surface hardness, compressive strength, and flexural strength of material close to that of enamel. Karimzadeh *et al*.[18] concluded in their study that there is influence of fillers on elasticity and hardness properties of nano composites. Z350 exhibited almost similar results in their study. Abouelleil *et al*.[11] in their study concluded that EverX™ exhibited better hardness values; this is in agreement with our study.

Conclusion

In the present study, it can be concluded that the mean compressive, flexural, and surface hardness values of all the five materials were significantly different because the composite materials available have variation in composition and viscosity. The nanohybrid composite CFM™ has the highest hardness and properties of flexural strength, flexural modulus, and the hardness of EverX™ and Z350 was almost similar. Compressive strength value of EverX™ was high compared with that of the other four composites. SDR™ exhibited the least values when compared with that of the other composites. In the present study, the null hypothesis was not accepted; further clinical studies should be carried out as all nanocomposites exhibited different mechanical properties.

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Conflicts of interest

There are no conflicts of interest.

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