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Properties of a Dental Resin Composite with a Spherical Inorganic Filler

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Clinical Relevance

Of the materials tested in this study, the spherical filler composite (Estelite Σ) had similar properties as the nano-composite (Filtek Supreme). Thus, Estelite Σ can be used in anterior regions and restricted posterior restorations. All the materials had a similar shrinkage pattern, in that about 99% of the shrinkage occurred prior to 24 hours; thus, for direct resin composite restorations, a strong initial bonding strength with bonding agent would be necessary.

SUMMARY

This study compared the mechanical properties, generalized wear resistance and polymerization shrinkage of a resin composite filled with spherical inorganic filler to other commercial resin composites. Six dental resin composites were tested, including a submicron filled composite (Estelite Σ, Estelite), 1 nano-composite (Filtek Supreme, Supreme), 2 microfilled composites (Heliomolar; Renamel Microfill, Renamel) and 2 microhybrid composites (Esthet X Improved; Tetric Ceram). Compressive strength (CS), diametral tensile strength (DTS), flexural strength (FS), flexural modulus (FM), generalized wear resistance (WV) and polymerization shrinkage (PS) were evaluated for the 6 materials. The specimens were cured according to the manufacturers' instructions in appropriate molds, stored (37°C water, 24 hours), then tested on an Instron testing machine (0.5 mm/minute). PS was tested according to the Archimedes method at 1, 24 and 48 hours continually after polymerization. Data were analyzed by analysis of variance. The results showed that CS values ranged from 252 to 298 MPa, DTS ranged from 35 to 54 MPa, FS from 73 to 140 MPa, FM from 4.8 to 11.1 GPa, WV from 0.037 to 0.086 mm^3 and PS at 24 hours from 2.17 to 3.96 vol%. Composite had statistically significant influence on the *in vitro* properties tested. Estelite performed similarly to nano-composite and microhybrid composites in mechanical properties and generalized wear resistance, while Estelite and Supreme had the lowest PS among the materials tested. The 2 microhybrid materials had similar properties, while the 2 microfilled composites were different for most properties tested. Overall, the microfilled composites had lower strength than the other composites except

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Renamel for CS. All the materials had a similar shrinkage pattern in that about 99% of shrinkage occurred in less than 24 hours.

INTRODUCTION

The ultimate goal of dental restorative materials is to replace the biological, functional and esthetic properties of healthy tooth structure. Dental amalgam and gold alloys, which have a long record of clinical success, have been used as dental restorative materials for more than 100 years, especially in posterior teeth, because their mechanical properties match those of natural teeth;[1-2] however, these metallic materials are not esthetic. Since their introduction into the dental market 40 years ago, dental resin composites have proven to be successful. It is expected that usage of resin composites in posterior teeth will continue to grow.[3]

Although considerable improvements have been made in the properties of dental resin composites over the years, no fundamental change in monomer systems has occurred since Bowen introduced dimethacrylates in the form of Bis-GMA in 1962. Major developments come from improvements in filler systems.[4-5] Resin composites have gone through generations of traditional (macrofilled) composites, microfilled composites, hybrid composites, microhybrid composites and nano-composites. Filler loading has been shown to correlate with the material's strength, elastic modulus, wear resistance and polymerization shrinkage,[6-10] whereas filler size influences the restoration's polishability.[11-13] The average filler size in microhybrid composites has been reduced to less than 1 micron to achieve polishability, while retaining higher filler loading. A certain filler size distribution is usually necessary to get high filler loading in microhybrid composites.[14-15] Fillers in most microhybrid composites are ground glass particles whose morphology is irregular (Figure 1). Nano-composites are a relatively new generation of composites. For example, Filtek Supreme has a combination filler system that consists of nanomeric particles and nanoclusters. Its primary fillers are 20 nm or 75 nm chemically synthesized spherical silica, while the nanoclusters are around 0.6 μm.[12]

A new type of filler has been developed for Estelite Σ (Estelite) by Tokuyama Dental Corp. The filler produced by the sol-gel method has a spherical shape (Figure 2). The average particle size is 0.2 μm, with a narrow range from 0.1 to 0.3 μm, thus, the material is called a submicron composite.

This study compared mechanical properties, generalized wear resistance and polymerization shrinkage of Estelite, a spherical shape filler incorporated resin composite, to 5 popular contemporary resin composites.

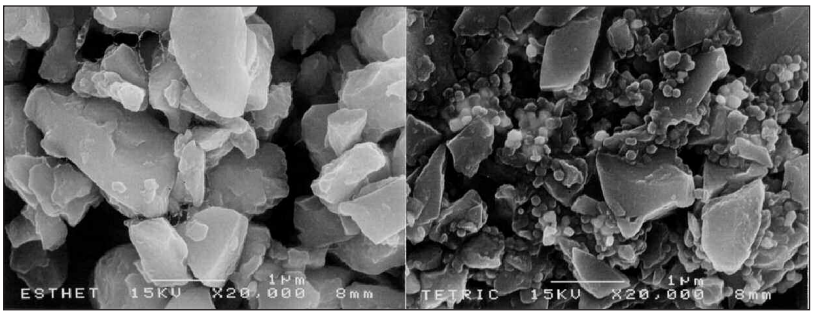

Figure 1. *Irregular inorganic filler particles in microhybrid composites (left: Esthet X Improved; right: Tetric Ceram).*

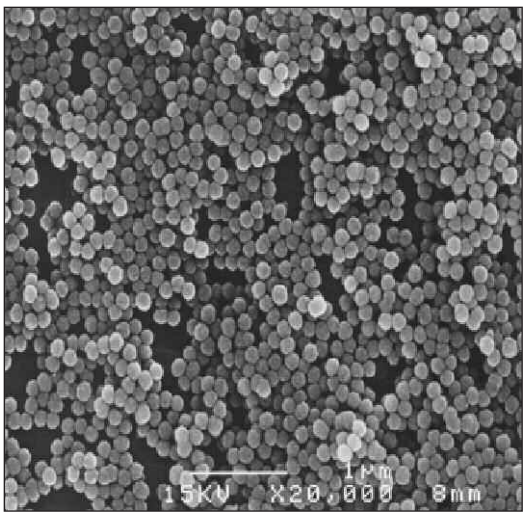

Figure 2. *Spherical fillers in Estelite* Σ.

The mechanical properties tested were compressive strength, diametral tensile strength, flexural strength and flexural modulus.

METHODS AND MATERIALS

The 6 dental resin composites used in this study are listed in Table 1, including 1 submicron filled composite–Estelite; 1 nano-composite–Filtek Supreme (Supreme); 2 microfilled composites–Heliomolar, Renamel Microfill (Renamel) and 2 microhybrid composites–Esthet X Improved (Esthet X), Tetric Ceram.

For compressive strength (CS), the materials were condensed into a split polytetrafluoroethylene mold (8 mm in length and 4 mm in diameter) covered with a Mylar strip and a glass microscope slide. After the top and bottom surfaces of the specimen were light cured per the manufacturer's instructions, the specimen was taken out of the mold and light cured in the middle of the specimen at opposing sides. A curing unit (Elipar Highlight, 3M ESPE, St Paul, MN, USA) with 10-mm optic diameter was used. The intensity of the curing light (500 mW/cm^2) was monitored with a radiometer (Kerr/Demetron, Danbury, CT, USA).

Table 1: *Resin Composites Tested in This Study*

Brand Name and Classification	Manufacturer	Composition	Curing Time (seconds)
Estelite Σ, Submicron filled composite	Tokuyama Dental Corp, Tsukuba, Japan	Filler: 71 vol.% (82 wt.%) of spherical silica-zirconia filler of 0.1-0.3 μm (average, 0.2 μm) and prepolymerized filler of silica-zirconia and copolymer; Resin: Bis-GMA, TEGDMA	30
Filtek Supreme, Nano-composite	3M ESPE, St Paul, MN, USA	Filler: 59.5 vol.% (78.5 wt.%) of combination of nanomeric particles and nanoclusters with primary particle size of 20 or 75 nm Resin: Bis-GMA, Bis-EMA(6), UDMA, TEGDMA	20
Heliomolar, Microfilled composite with radiopacity	Ivoclar Vivadent, Amherst, NY, USA	Filler: 46 vol.% (77.8 wt.%) of highly-dispersed silicon dioxide, ytterbium trifluoride and copolymer of 0.04-0.2 μm Resin: Bis-GMA, UDMA, decandiol dimethacrylate	40
Renamel Microfill, Microfilled composite	Cosmedent, Chicago, IL, USA	Filler: 60 wt.% pyrogenic silicic acid filler of 0.02-0.04 μm Resin: multifunctional methacrylate ester	60
Esthet X Improved, Microhybrid composite	DENTSPLY Caulk, Milford, DE, USA	Filler: 60 vol.% of barium boron fluoroalumino silicate glass (irregular) with an mean particle size below 1 μm and nanofiller silica (0.04 μm) Resin: Urethane modified Bis-GMA dimethacrylate	20
Tetric Ceram, Microhybrid composite	Ivoclar Vivadent Amherst, NY, USA	Filler: 60 vol.% (82.6 wt%) filler of 0.04-3.0 μm (average 0.7 μm) (combination of irregular and spherical fillers) Resin: Bis-GMA, UDMA, TEGDMA	20

BIS-GMA (Bisphenol A diglycidyl ether dimethacrylate); TEGDMA (tri[ethylene glycol] dimethacrylate); Bis-EMA(6) (Bisphenol A polyetheylene glycol diether dimethacrylate); UDMA (urethane dimethacrylate)

For diametral tensile strength (DTS), the materials were packed into a split polytetrafluoroethylene mold (6 mm in diameter and 3 mm in depth), covered with a Mylar strip and a glass microscope slide and light-cured at the top and bottom surfaces.

For flexural strength (FS) and modulus (FM), a stainless steel split mold was used to prepare the flexural specimens with a dimension of 2 mm x 2 mm x 25 mm. The material was packed into the mold, and the specimen was light cured in 5 overlapping sections to ensure maximum conversion on both the top and bottom surfaces, according to ISO 4049.[16]

The specimens (n=5) were stored in 37°C distilled water for 24 hours before being tested on a universal material testing machine (Instron 4465, Instron Corp, Canton, MA, USA) at a crosshead speed of 0.5 mm/minute. Dimensions of the specimens were determined by a digital caliper (Absolute Digimatic, Mitutoyo Corp, Tokyo, Japan). CS, DTS, FS and FM values were determined by software (Series IX, version 8.32.00, Instron Corp, Norwood, MA, USA).

Generalized wear resistance was tested with a Leinfelder-type wear tester for 400,000 cycles. Composite was filled into a cavity (8 mm in diameter and 3 mm in depth) on resin block in 2 increments and light cured. The top layer was covered with a Mylar strip, and a glass slide was used to gently expel the extra material before light curing. Prior to testing, the specimens were stored in distilled water at 37°C for 24 hours. Eight specimens were made for each resin composite.

A tight fitting metal ring filled with water slurry of non-plasticized PMMA beads (HG-5, Dentsply/Caulk, mean bead size: 44 μm) surrounded the mounted specimen. The mixing ratio was 15 g PMMA to 9 ml distilled water. A flat stylus (with a diameter of 6 mm) made with polyacetal was perpendicularly loaded onto the center of the specimen at a rate of 2 times per second under a load of 76-80 N. The piston load was checked every 100,000 cycles of wear. Upon contacting the specimen's surface, the stylus began to rotate 30 degrees. After achieving the maximum load, the stylus initiated counter rotation and moved in an upward direction. The entire cycling procedure was carried out 400,000 times. The amount of wear, wear volume (WV), was measured with a 3-dimensional profilometer (MTS, St Paul, MN, USA), which was the total wear-off volume.[17]

Polymerization shrinkage (PS) was tested according to the Archimedes method at 1, 24 and 48 hours continually after polymerization. About 0.4-0.5g of paste was weighted in air and water, respectively, on an analytical electronic balance (A-160, Fisher Scientific, Hampton, NH, USA) and a density kit (YDK01, Sartorius Corp, Edgewood, NY, USA). The paste was then cured for 90 seconds each on both sides in a light-curing chamber (UniXS, Heraeus Kulzer, Armonk, NY, USA). After storing in air for 1 hour at room temperature, the specimen was re-weighted in air and water, respectively. The

same specimen was then stored dry at 37°C and re-weighted at 24 and 48 hours after polymerization.

Volumetric shrinkage was calculated from density using the Archimedes method using the following formula:

$$PS = [1 - \frac{(W_{1a} - W_{1h})/\rho_{1h}}{(W_{2a} - W_{2h})/\rho_{2h}}] \times 100\%$$

where W stands for weight, ρ for water density, the subscripts $_1$ and $_2$ represent the un-polymerized paste and cured material, respectively, while $_a$ and $_h$ represent in air and water, respectively.

Statistical Analysis

Data were analyzed by 1-way analysis of variance (ANOVA, SAS 8.0, SAS Institute, Cary, NC, USA) to detect the influence of composite on properties. Fisher's protected least significance difference intervals (Fisher's PLSD) were calculated at the 0.05 level of significance (α=0.05) to compare the means among composites. NCSS (NCSS/PASS Dawson Edition, Number Cruncher Statistical Systems, Kaysville, UT, USA) was used to test ANOVA assumptions. The ANOVA assumption of independence was met as the specimens were made individually.

RESULTS

Table 2 lists means and standard deviations of the tested properties, including CS, DTS, FS, FM, WV and PS. Note that for CS, DTS, FS and FM, the higher the value, the stronger the composite. However, for WV and PS, the lower the value, the better the property. The 2 microhybrid composites had similar properties, while the 2 microfilled composites were different for most properties tested. Overall, the microfilled composites had lower mechanical strength than other composites except Renamel for CS. Submicron filled Estelite and nanofilled Supreme had similar results for most of the properties tested. All the materials had a similar shrinkage pattern, in that about 99% of the shrinkage occurred prior to 24 hours. Estelite and Supreme showed the lowest PS among the materials tested, while Renamel showed the highest.

Normality and equal variance assumptions of ANOVA were met for the data of CS, DTS, FS, WV and PS24, while normality assumption was minor violated for FM. As ANOVA was relatively robust to minor assumption violation, this method was used to test the effect of composite on all those properties. Composite had a statistically significant influence on all the properties. The materials that were statistically different are identified by superscript letters in Table 2.

DISCUSSION

Mechanical Properties

CS, DTS and FS are measures of the strength of material under different force conditions. The higher the value, the stronger the material. ISO 4049 classifies dental polymer-based restorative materials into 2 types: Type I is the material claimed by the manufacturer to be suitable for restorations involving occlusal surfaces, and Type II are all other polymer-based filling and restorative materials. The minimum flexural strength requirement for Type I is 80 MPa and 50 MPa[16] for Type II. The results of this study showed that all the composites tested had a flexural strength higher than 80 MPa except Renamel, whose FS was 73 MPa. As a microfilled composite, Renamel was intended to be used in the anterior region, so it should be categorized as Type II material. Thus, Renamel met the ISO requirement as well. However, the scientific rational and clinical relevance for the minimal values defined by ISO 4049 are not clear; ISO specification can only be used for quality control. On the other hand, as testing conditions such as temperature and crosshead speed have significant influence on the values of the mechan-

Table 2: *Means and standard deviations of compressive strength (CS), diametral tensile strength (DTS), flexural strength (FS), flexural modulus (FM), wear volume (WV) and polymerization shrinkage (PS) of the tested resin composites.*

	CS (MPa)	DTS (MPa)	FS (MPa)	FM (GPa)	WV (×10^{-2} mm^3)	PS (vol%) 1 Hour	PS (vol%) 24 Hours	PS (vol%) 48 Hours
Estelite Σ	297 (35)^{ab}	47 (3)^{de}	112 (14)^{hi}	7.0 (0.2)	3.7 (2.5)^{m}	1.65 (0.27)	2.16 (0.23)^{o}	2.17 (0.28)
Filtek Supreme	262 (20)^{c}	54 (4)^{d}	140 (13)^{j}	11.1 (0.5)	5.4 (2.1)^{m}	1.68 (0.25)	2.26 (0.23)^{o}	2.27 (0.22)
Heliomolar	252 (41)^{c}	35 (6)^{f}	101 (9)^{i}	5.9 (0.2)	8.6 (3.7)^{n}	1.96 (0.11)	2.54 (0.18)	2.52 (0.19)
Renamel Microfill	298 (15)^{a}	38 (8)^{fg}	73 (6)	4.8 (0.1)	6.2 (3.4)^{mn}	3.24 (0.14)	3.97 (0.10)	3.96 (0.15)
Esthet X	263 (25)^{bc}	46 (9)^{eg}	125 (12)^{hk}	8.9 (0.1)^{l}	6.5 (3.6)^{mn}	2.60 (0.22)	3.09 (0.23)^{p}	3.10 (0.25)
Tetric Ceram	263 (7)^{bc}	49 (3)^{de}	134 (9)^{jk}	9.2 (0.3)^{l}	4.1 (2.7)^{m}	2.56 (0.23)	3.28 (0.09)^{p}	3.26 (0.16)

Means with same superscript letters were not statistically different at the 0.05 level of significance. (n=5, except n=8 for WV).

ical properties of resin composites, more clinically relevant testing methods are needed.[18]

Flexural modulus describes stiffness, a measure of the resistance to deformation under load of the material, with a high number indicating greater stiffness. There are debates on how much modulus resin composites should possess.[19-20] The ideal value should be similar to that of tooth structure, so that the restoration could have similar deformation with the surrounding tooth structure under load. When compared to the moduli of human enamel and dentin, which are about 84 GPa[21] and 14 GPa[22], respectively, resin composites had much lower values. In contrast, dental amalgam and gold, whose moduli are about 50 GPa[23] and 90 GPa,[24] respectively, have successfully served as posterior restorative materials for quite some time.[1-2]

Although there were statistically significant differences among materials for CS, the relative differences among materials were not as large as other properties, and it is not clear whether the difference has clinical significance. Overall, microfilled materials (Heliomolar and Renamel) had lower DTS, FS and FM than other materials. These lower properties can be explained by their lower filler loadings (Table 1). Estelite, which has spherical submicron filler, had comparable mechanical properties to the nano-composite and microhybrid composites tested.

Wear

The Leinfelder wear tester can be used to simulate 2 types of clinical wear mechanisms. The wear provoked by a food bolus during the masticatory process is simulated by the generalized wear test,[25] and the occlusal contact wear created by antagonistic cusps is simulated by the localized wear test.[26] Although dental resin composites have been used extensively in posterior teeth, it is recommended that they be used in small to medium size cavities, and not extensive restorations, in order to reduce direct occlusal contact.[27] As a result, a modified generalized wear test was used in this study. A flat-planed stylus with a diameter of 6 mm and made from polyacetal was used, whereas, the diameter of the resin composite specimen was 8 mm. The load from the stylus was fully applied to the specimen, which created a wear model between the traditional generalized and localized wear. The traditional generalized wear test uses an 8.0 mm diameter flat stylus, and the composite specimen has a diameter of 4.0 mm and is filled in an enamel cavity, so that the stylus completely covers the restoration and 2 mm of the adjacent enamel surface.[25,28]

As the standard deviation for the WV data was large, statistical analysis showed that only Heliomolar had significantly higher wear volume than Estelite, Supreme and Tetric Ceram, while no differences were detected among Estelite, Supreme, Renamel, Esthet X and Tetric Ceram (Table 2). The results of this study were contrary to Leinfelder's findings in that Heliomolar had lower wear depth than the microhybrid composites.[25] The difference in test procedure and characterization of the amount of wear might explain the disparity between studies. Heintze and others showed that Heliomolar demonstrated poor global performance when tested with different wear simulators.[29] They concluded that the different wear simulator settings measured different wear mechanisms. It seems reasonable to combine at least 2 different wear simulations to assess the wear resistance of a new material. As for mechanical properties, Estelite showed comparable wear resistance to most of the other materials tested.

Resin composites from different classifications have different wear mechanisms that are influenced mostly by filler systems in the material, as the resin systems used in today's resin composites are similar. Microfilled composites and nano-composites have small primary filler particles such that the filler and resin matrix are abraded off together during wear. For microhybrid composites whose average filler particle sizes are approximately 1 μm, the relatively soft resin matrix is worn first, and the inorganic filler stands above the surface. When there is not sufficient resin around the filler, the individual filler particle is plucked out, leaving a void on the surface and a new wear cycle begins.[12,30] Inorganic fillers with higher hardness can help reduce the restoration's wear,[9,31] but, on the other hand, they increase the surface roughness of restorations on the occlusal contact areas and can cause excessive wear of the opposing enamel.[32] In contrast, spherical filler might keep the surface smoother than irregular shaped filler after it is exposed to the surface. In another study, Estelite maintained the highest gloss during wear among the materials tested.[33] Spherical filler might help to reduce friction during wear, and it may be kinder to the opposing dentition than microhybrid composites. For similar particle sizes, an irregularly-shaped filler particle composite was more wear-resistant than the spherically-shaped filler materials. Spheres will probably not provide any mechanical retention between the filler and resin matrix, and irregularly-shaped filler offers higher specific surface area for adhesion.[34] In this study, spherical filler composites showed similar wear resistance to that of microhybrids.

The results of this study agree with other studies in that wear resistance did not correlate with other mechanical properties tested.[35-36]

Polymerization Shrinkage

Despite improvements in mechanical properties and the wear resistance of resin composites over the years, polymerization shrinkage has been an inherent deficiency of dental resin composites. The clinical consequences of polymerization shrinkage, such as secondary caries, marginal discoloration and postoperative sensi-

tivity, are the main reasons for replacement of resin composite restorations.[37-41] Although non-shrinking resin composites have been studied for many years, no such products are available in today's dental market.[4,42]

Volumetric polymerization shrinkage is mainly determined by composition of the material, such as the type and amount of the resin matrix used, the initiation system and filler loading.[43-44] The degree of conversion of the resin system has a direct relationship with polymerization shrinkage.[45-46] The results of this study showed that all the materials had a similar shrinkage pattern, in that about 99% of shrinkage occurred prior to 24 hours. The values of PS24 and PS48 were nearly the same (Table 2); therefore, PS24 was chosen for the statistical analysis. Estelite and Supreme had a similar PS, followed by Heliomolar, while Renamel showed a significantly higher PS among the materials tested. The low filler loading in the microfilled composite Renamel might explain its high PS. The other microfilled composite (Heliomolar) was filled with prepolymerized resin fillers, which served to reduce the amount of unpolymerized resin matrix. Although there were significant differences in PS24 for Estelite and the microhybrid composite materials, filler geometry did not seem to influence the degree of conversion of resin composites;[34] thus, it probably would not influence polymerization shrinkage. The smaller specific surface areas of spherical fillers need less resin matrix to wet them and, thus, allow for achieving high filler volume loading in Estelite, which contributed to the low PS value.

CONCLUSIONS

Estelite behaved similarly to nano-composites and microhybrid composites in mechanical properties and generalized wear resistance, while Estelite and Supreme had the lowest PS among the materials tested. The 2 microhybrid materials had similar properties, while the 2 microfilled composites were different for most properties tested. Overall, the microfilled composites had lower strength than other composites except Renamel for CS. All the materials had similar shrinkage patterns, in that about 99% of the shrinkage occurred before 24 hours.

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