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Characterization of third-body media particles and their effect on *in vitro* composite wear

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Abstract

Objectives—The purpose of this study was to compare four medium particles currently used for *in vitro* composite wear testing (glass and PMMA beads and millet and poppy seeds).

Methods—Particles were prepared as described in previous wear studies. Hardness of medium particles was measured with a nano-indentor, particle size was measured with a particle size analyzer, and the particle form was determined with light microscopy and image analysis software. Composite wear was measured using each type of medium and water in the Alabama wear testing device. Four dental composites were compared: a hybrid (Z100), flowable microhybrid (Estelite Flow Quick), micromatrix (Esthet-X), and nano-filled (Filtek Supreme Plus). The test ran for 100,000 cycles at 1.2Hz with 70N force by a steel antagonist. Volumetric wear was measured by non-contact profilometry. A two-way analysis of variance (ANOVA) and Tukey's test was used to compare both materials and media.

Results—Hardness values (GPa) of the particles are (glass, millet, PMMA, poppy respectively): 1.310(0.150), 0.279(.170), 0.279(0.095), and 0.226(0.146). Average particle sizes (μm) are (glass, millet, PMMA, poppy respectively): 88.35(8.24), 8.07(4.05), 28.95(8.74), and 14.08(7.20). Glass and PMMA beads were considerably more round than the seeds. During composite wear testing, glass was the only medium that produced more wear than the use of water alone. The rank ordering of the materials varied with each medium, however, the glass and PMMA bead medium allowed better discrimination between materials.

Significance—PMMA beads are a practical and relevant choice for composite wear testing because they demonstrate similar physical properties as seeds but reduce the variability of wear measurements.

Keywords

Composite wear; third-body medium; dental composite; hardness

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Introduction

While wear of dental composites is a prolifically studied subject [1], there is great variability in testing methods [2]. The 2001 International Standards Organization report “Wear by two and or three body contact” describes eight methods for measuring *in vitro* wear. Among other variables, the report describes three different food-simulating media for three-body wear including: millet seed, PMMA beads, and poppy seed [3]. The adoption of these media particles originated from a 1986 publication from de Gee, who compared wear rates produced with different seeds and polymethyl methacrylate (PMMA) powder in the ACTA wear testing device. He determined that using a mixture of 80% millet seeds and 20% PMMA powder most closely correlated *in vivo* wear data [4]. Later, Leinfelder and Suzuki used PMMA alone as a third-body medium because it does not degrade like millet and also expedites the wear process [5,6]. Condon and Ferracane introduced poppy seed as a replacement to millet seed in de Gee's original mixture [7,8]. Since that time, additional third body particles have been examined including hydroxyapatite, green carborundum [9] and calcium diphosphate [10]. Glass microbeads have also been used as a third-body medium in industry protocol to expedite wear testing.

Although the effect of media particle selection on composite wear has not been directly studied, various test methods which incorporate different particles have been compared. Two studies by Heintze et al compared the ACTA, Alabama and OHSU wear testing methods, which incorporate millet seed, PMMA, and poppy seed media respectively. These studies determined that relative wear ranking of composite materials varied significantly between testing methods [11,12]. Among those testing methods, there is variation in many other factors (such as the methods of masticatory force application and tooth sliding reproduction) [13], so it is not possible to attribute the discrepancy in wear ranking to variation in media particles alone. The aim of this study is to compare the wear of four composites in the Alabama wear testing device with four currently used third-body medium particles (millet seed, poppy seed, PMMA beads, and glass microbeads) and water. The null hypothesis is that the ranking of materials will be similar for all medium used.

Measuring the physical properties of the abrasive particles is critical for understanding the wear-producing mechanisms that differentiate each medium. Theoretically, a particle will be more abrasive if: 1. it is harder than the surface it is indenting and 2. the size and form of the particle allow it to penetrate through composite filler particles to the wear-prone resin matrix. The hardness, size and shape of each abrasive medium particle will be measured in this study, as these properties have been identified as critical parameters in tribological testing [14].

Materials and methods

Media particle preparation

Millet seed was prepared, as described by Nihei [15], by grinding 50g of seeds in a rotating blade grinder for 5 seconds. Poppy seed was prepared as described by Condon and Ferracane [7] by grinding 3g of poppy seed with 100 strokes of mortar and pestle. PMMA beads (Dentsply Caulk, Milford, DE, USA) and soda lime glass microbeads (Size 270, Unibrite Corporation, Port Washington, NY, USA) were obtained from their manufacturer.

Nano-hardness measurement

The medium particles were embedded in a 95% methyl methacrylate / 5% nButyl embedding epoxy (Fischer Scientific, Pittsburgh, PA, USA) before testing. The glass and PMMA beads were first stained with methylene blue to aid in their visualization. A thin coat of each type of medium particle was dispersed on the surface of a cup half-filled with set

epoxy. The specimens were then covered with a layer of unset epoxy which polymerized under ultraviolet light for 48 hours. The surface of the specimens were wet polished with a succession of 320 grit, 800 grit and 1200 grit paper on a surface parallel plane grinder (400CS, Exakt Technologies Inc, Oklahoma City, OK, USA) to reveal a layer of sectioned particles. The nano-hardness of the exposed surfaces of the medium particles was measured with a nano-indentation tester (G200, MTS, Oak Ridge, TN, USA). Indentations were depth controlled to 0.5 μ m and performed with a diamond Berkovitch pyramid-shaped stylus (diameter = 40nm). A 4 \times 4 grid of indents (5 μ m spacing between indents) was selected on three millet and poppy seeds. Fifteen individual glass and PMMA beads were selected for testing. Indents were examined after testing and hardness values that were obtained from indenting the epoxy were discarded.

Composite specimens were prepared in a silicone mold (1cm diameter \times 4mm) and light polymerized at 2mm increments with a Coltolux LED curing light (Coltene/Whaledent, Cuyahoga Falls, OH, USA) (583mW/cm²). They were then polished using 600 and 1200 grit silicon carbon paper followed by 0.5 μ m alumina slurry on a polishing wheel (Metallurgical polisher, Buehler Ltd., Evanston, IL, USA) at 80 rotations/sec and 20N of force. Nano-hardness of the composites was determined by creating a 4 \times 4 grid of indents (5 μ m spacing between indents) at two locations on the composite surface. The same testing parameters were used as described above.

Particle size measurement

The medium particles were mixed with distilled water in a 3:1 ratio. A 3mL sample of each medium was measured in a LASER light diffraction optical particle size analyzer (Microtrac 3500, Microtrac Inc., York, PA, USA) operated between the size range of 24nm and 2800 μ m. Three measurements were taken of each sample, and media were sonicated for 2 minutes between measurements to prevent agglomeration.

Particle imaging and shape measurement

Particles were randomly dispersed on a glass slide. The particles were imaged with 1000 \times optical magnification using digital light microscopy (VHX-600, Keyence Co., Osaka, Japan) as described in Table 1 of ASTM standard F1877-05 [16]. Three images of each medium were collected, and within the images, the perimeter (P) and area (A) of the outline of each particle was recorded with image analysis software (ImageJ, NIH, Bethesda, MD, USA).

The form of the particles was determined using the form factor (FF) equation: $FF = \frac{4\pi A}{p^2}$ [14]. Form factor gives an indication of the roughness or roundness of a particle's outline; particles with a circular outline have a FF = 1. The average of the form factor of all particles from each media type was reported.

Wear testing of composite materials

Four commercially available light-cured composites were studied: a hybrid (Z100, 3M Co., St Paul, MN, USA), a flowable microhybrid (Estelite Flow Quick, Tokuyama, Tokyo, Japan), a micromatrix (Esthet-X, Caulk Dentsply, Milford, DE, USA), and a nano-filled (Filtek Supreme Plus, 3M ESPE). The materials were chosen to represent a range of filler concentrations (71%-85%) and filler particle sizes (0.005-3.5 μ m). Their properties are listed in Table 1.

Specimens (n=8) were fabricated in silicone molds (1cm diameter \times 4mm depth) and light polymerized at 2mm increments with a Coltolux LED curing light (Coltene/Whaledent) (583mW/cm²). After production, the specimens were set in brass holders with acrylic (Dentsply Repair Material, Dentsply Caulk) and polished using 600 and 1200 grit silicon

carbon paper followed by 0.5 μm alumina slurry on a polishing wheel (Metallurgical polisher, Buehler Ltd.) at 80 rotations/sec and 20N of force. Specimens were stored in water at 37°C for 48 hours. The spring containing antagonist pistons were load calibrated before testing with a universal testing device (Model 4411, Instron, Norwood, MA, USA) to ensure a maximum force of 70N. Specimens were placed into the Alabama wear device, which operates by pressing the spring-loaded pistons into the composite specimens followed by a 30° rotation of the antagonist. The antagonists on the pistons contact the specimens for 400ms applying a maximum force of 70N and counter-rotate 30° prior to lifting off the specimens (Figure 1). 2g of the medium prepared as described above was mixed with 3mL of distilled water. This ratio was chosen based on previous studies [4,15]. The medium mixture was stirred and vibrated until the particles fully mixed with the water. Due to the differences in densities of each type of particle, some wells contained more particles than others, however, all specimens were completely covered with medium. The medium was then poured into the individual wells above the composite specimens created by the brass rings (Figure 2). A fifth group was prepared in which the specimen wells were filled with distilled water. The test was run for 100,000cycles at 1.2Hz. A new stainless steel antagonist ball ($R_a = 4.7\mu\text{m}$) was used for every test. Following the test, specimens were ultrasonically cleaned for 1 minute. The surface of each specimen was scanned with a non-contact optical profilometer (Scantron 2000, Scantron Industrial Products, Tauton, England) with a 20 μm \times 20 μm resolution. The scans were analyzed with superimposition software (Pro-Form, Scantron Industrial Products) to determine volumetric wear.

Following testing, representative wear specimens were removed from the brass holders and coated with Au-Pd in a sputter coater (Hummer X, Anatech, Union City, CA, USA). Their surfaces were examined by secondary-electron SEM (Model 40, International Scientific Instruments, Milpitas, CA, USA). Samples of glass and PMMA beads were removed from the wells following testing and examined using digital light microscopy (VHX-600, Keyence Co).

Statistical analysis

The study design was a two-way layout, with groups defined by material and medium. The primary analysis technique utilized two-way analysis of variance (ANOVA). Pairwise comparisons among group means were conducted using Tukey's test. A rank transformation of the data was used, due to significant nonhomogeneity of variance among the groups ($p < 0.0001$, Levene's Test), and substantial asymmetry of the sample distributions. Separate one-way ANOVA analyses were conducted for materials within each medium and medium within each material group in order to evaluate the significant interactions.

Results

The nano-hardness of the composite materials is given in Table 2. The mean, standard deviation and coefficient of variation (COV) of the nano-hardness, form factor and size for each type of particle is given in Table 3. The COV was calculated using the formula $\text{COV} = \text{mean}/\text{standard deviation}$. The COVs of nano-hardness, form factor and size values of the PMMA and glass beads were less than those of the millet and poppy seeds. The lower COV of PMMA and glass beads indicated a more homogenous composition of these particles. The size distribution of particles as distributed by number of particles is graphed in Figure 3. Images of the media particles are presented in Figure 4.

The mean, standard deviation, and coefficient of variance of volumetric wear data is given in Table 4. The results of a 2-way ANOVA revealed that there was a significant material by solution interaction ($p < 0.0001$), as well as significant main effects for material ($p = 0.004$) and solution ($p < 0.0001$). The individual analyses by medium type reveal that materials can

be grouped into significantly different groups for all medium types except millet seeds. The individual analyses by material type reveal that there was significantly more wear on all materials when glass was used as a medium than in water alone and that there was less wear on all materials when poppy seeds, millet seeds and PMMA beads were used as medium than in water alone (Table 4).

SEM images are presented for the worn surface of Filtek Supreme Plus as representative examples for all materials (Figure 5). The figure shows that wear track that is produced from the contact and 30° rotation of the spherical stainless steel antagonist. The wear track in the glass group (Figure 5A) shows deep gauging and spalling of the composite surface and it is larger than the tracks from all other groups. The edges of the wear track appear jagged from material that has chipped off from contact with the hard glass particles. The wear track on the water group shows deep, smooth gauging in the wear track (Figure 5E). The smoothness of the tracks from the water group compared to the glass group can be attributed to the smaller dimensions of the asperity heights on the steel antagonist (~4.7µm) than size of the glass beads (~88µm). The PMMA, millet and poppy groups (Figure 5 B-D) show superficial scratching of the composite surface. These scratches may have occurred from the three-body abrasion of medium particles or periods of direct two-body contact between the steel antagonist and the composite surface.

A micrograph of PMMA beads following wear testing (Figure 6) demonstrates surface roughening of several of the beads. Roughening was not noted on the glass beads and no difference could be visibly discerned between the seeds before and after wear testing (however degradation of the seeds is suspected).

Discussion

In vitro wear testing should predict the wear performance of a material *in vivo*. To date, only one study has reported a correlation between the amount of wear produced in the Alabama wear testing device and clinical wear measurements [17]. Due to limited clinical wear data, it is more practical to utilize *in vitro* wear testing to determine the relative rankings of materials than quantify an expected amount of wear. Another practical utility of an *in vitro* wear test is its ability to discriminate between materials. Variation among specimens should be attributed to the differences in the physical properties of the material not the variation in a component of the wear testing system, such as the food simulating medium. It is important, however, to include a food simulating medium in wear testing as previous studies have shown both increased and decreased wear in the presence of a third-body medium [18,19].

Based on the results of this experiment, material ordering with regard to wear was dependent on the medium used. Individual analyses by medium type revealed that significant differences could be seen between materials using glass, PMMA and poppy seeds but not millet seeds. Therefore, the null hypothesis cannot be accepted. Additionally, the use of glass beads produced more composite wear than with water alone and the use of all other medium particles resulted in less wear than the use of water alone. An analysis of the medium particle properties will be used to try to explain differences in relative wear values, discriminating ability of the different media, and material ordering.

The most obvious difference between medium particles was the hardness of the glass beads. A recent study examined the nano-hardness of several composite materials [20]. In that study, both the hardness of the resin matrix and filler particles were measured. The materials tested were similar to the materials tested in this study, containing both dimethacrylate resin matrix and silica-and zirconia-based filler particles. The reported resin matrix hardness ranged from 0.3-0.5GPa and the filler particle hardness ranged from 2-4GPa. Based on the

measured hardness of the medium particles in this study, the PMMA, millet and poppy particles are slightly softer than the resin matrix and much softer than the filler particles of composite materials. The glass beads, however, are much harder than the resin matrix and only slightly softer than the composite filler particles. The ability of the glass beads to abrasively wear the resin matrix and possibly even the filler particles of the composites explains the relatively large amount of composite wear using this abrasive medium.

The size and shape of abrasive particles and the interparticle spacing in a dental composite will determine the ability of a medium particle to penetrate to the resin matrix of a composite [21]. Jorgensen postulated that an interparticle spacing of less than $0.1\mu\text{m}$ would allow filler particles to protect the resin matrix from food abrasion. His theory was based on clinical observations of composite restorations [22]. Bayne et al theorized a method for determining the interparticle spacing of microfilled composites based on the volume fraction of filler particles. His calculations are based on $.02\mu\text{m}$ filler particles that are either evenly dispersed or agglomerated in resin matrix. The theorized maximum inter-particle or inter-cluster spacing was determined for various filler volume fractions [23]. The materials in this study range in volume fraction of filler from 53%-66% and particle sizes range from $0.01\text{--}3.5\mu\text{m}$, however, all materials contain some particles below $0.02\mu\text{m}$. Assuming all materials contain at least 10% vol of filler particles smaller than $0.02\mu\text{m}$, the theoretical maximum interparticle spacing would still be below $1\mu\text{m}$. Therefore, abrasive particles larger than $1\mu\text{m}$ would not be expected to penetrate through the interparticle spacing.

The mean size of all medium particles used in this study are larger than $1\mu\text{m}$, however, it is plausible that asperities on the surfaces of the medium particles could penetrate a $1\mu\text{m}$ space. Asperities are projections from a surface and the amount of asperities on the surface of a particle can be approximated by measuring its form factor. The PMMA and glass particles are spherical and do not have asperities (form factor of 1). The millet seeds and poppy seeds, however, have rough surfaces (form factor of 0.68 and 0.70 respectively) with asperities capable of penetrating through the interparticle spacing. The roughness of the seed particles may explain why most composites showed more wear with seed particles than PMMA beads, despite all three having similar hardness values. Micrographs of worn PMMA showed roughening of its surface, which may have created asperities and gradually increased the abrasivity of the PMMA beads.

The rank ordering of materials varied based on the type of medium particle used, however, due to the multifactorial nature of the wear processes and broad differences in materials, explaining the exact reasoning for the different rank orders is difficult. Several generalities regarding the wear rankings can be proposed based on the physical and mechanical properties of the composite materials and medium particles. Using glass as a third-body medium, the amount of composite wear was inversely related to the hardness and percentage filler content of the composite materials. This paradoxical relationship is explained by the fact that the glass beads have hardness values greater than the average bulk hardness of all the composites tested. The glass beads were twenty times the size of the largest filler particles and might have seen the composite surface as a bulk material. So even if the glass beads were not harder than the individual composite filler particles, they may have been able to fracture off asperities of bulk composite material. Since the glass was harder than the bulk surface of all composite materials, it could non-specifically wear the filler particles and resin matrix regardless of their hardness or percentage filler content. Therefore, the hardness of the composites relative to each other would not be useful for discriminating between materials. This theory is substantiated by the fact that SEM images of the worn composite shows gouging and spalling of the composite deep into the bulk of the material.

When PMMA was used as a third-body medium, the wear ranking was approximately related to the hardness and percentage filler content of the composite materials. This relationship suggests that wear testing with PMMA is affected by contribution of filler particles in the dental composite. The average PMMA particle size was about ten times the size of the largest filler particles. Therefore, it is unlikely that the entire PMMA bead penetrated the inter-particle composite spacing and selectively wore the resin matrix. The PMMA beads are softer than the bulk composite materials and much softer than the composite filler particles. During the wear process, the composite filler particles likely abraded the PMMA beads. Micrographs of the PMMA beads at the end of wear testing show that their surface becomes roughened. Possibly, the asperities on the roughened PMMA surface penetrated the interparticle spacing and preferentially wore the resin matrix. Therefore, composites with lower filler content would be less able to protect against this preferential wear. SEM images of the composite worn by PMMA media showed scratching of the composite surface, supporting the theory of preferential matrix wear.

The use of millet and poppy seeds in the third-body medium provided limited ability to discriminate between materials. The variation in wear volumes between specimens of the same material and seed type is evident by high values of the coefficient of variation. High COV is also present in the size and shape measurements of millet and poppy seeds. Since seed particles were stored in individual wells above each specimen during wear testing, the distribution of sizes and shapes of seed particles was different for every specimen. This variation in seed particles affected the abrasivity of the medium for each specimen and created variability in the resulting wear. Another source of variation using the seed particles is the differential breakdown of individual seed particles through mechanical deformation and water degradation. As the seed particles degrade through the wear process, they will become reduced in dimension. A study by Pallav et al showed that as medium particles are reduced from 3 μ m to 1 μ m, direct contact occurs between a composite and antagonist material [24]. Another study by Kunzelmann showed that larger crushed millet particles are more abrasive than smaller particles [25]. Therefore, the abrasive or lubricant properties of a batch of seeds are dependent on the individual breakdown of the seed particles in that batch. The tendency for the seed particles to degrade and allow direct two-body wear between the composite and the antagonist may explain the relatively higher wear with seen with a seed medium than a PMMA medium. SEM evaluation of the composite worn with millet and poppy seeds reveals scratching of the composite surface, similar in appearance as the surface worn by PMMA beads. These scratches may either be a result of harder seed particles scratching the resin in the composite or direct contact of the steel antagonist through degraded seed particles.

A limitation of this study was that it was difficult to explain differences between composite materials due to the compositional variability of commercially available materials. More materials would need to be included in this analysis to make a definitive conclusion of the most relevant third-body medium particle. Future studies should consider using materials standardized for resin and filler composition and filler particle size and size distribution to better understand the wear process. An additional limitation of this study is that these results are only applicable to the Alabama wear testing device and the sizes of PMMA and glass beads used in this protocol.

Conclusion

Based on the analysis of medium particles and composite wear, the PMMA used in this study seems to be a practical and relevant material to use in a third-body medium for composite wear testing. It has a similar hardness as natural seed materials. It is slightly larger than seed particles, however, the size distributions have overlap. The seeds are

rougher than the PMMA initially, but PMMA roughens through the wear process. Composite wear using a PMMA abrasive produces volumetric wear on the same order of magnitude as wear with natural seeds. Finally, SEM evaluation of wear surfaces appear similar with PMMA and natural seeds. The advantage of using PMMA as a third-body medium is that it produced less variation between specimens of the same material than seeds and therefore a greater ability to discriminate between different materials. For these reasons, the authors recommend using the PMMA used in this study for *in vitro* three-body wear testing.

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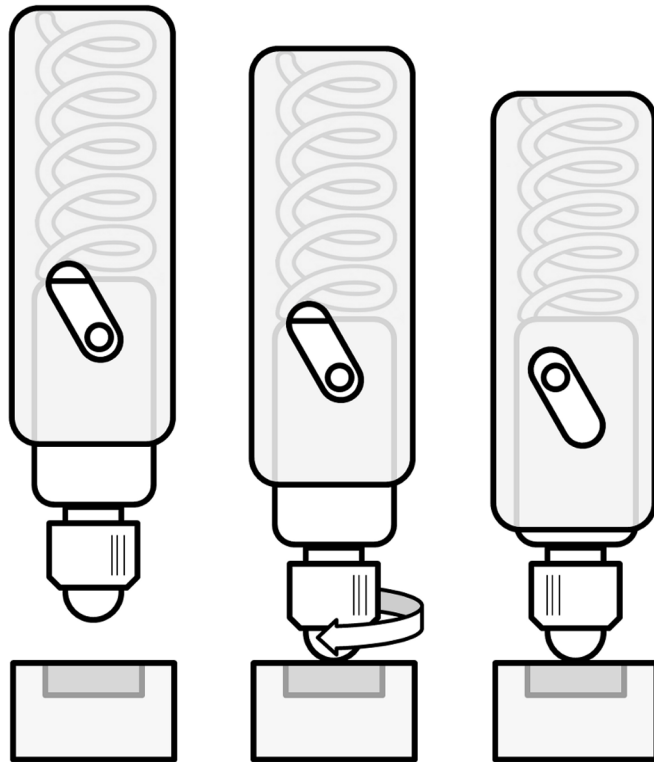


Figure 1. Schematic of Alabama wear testing device

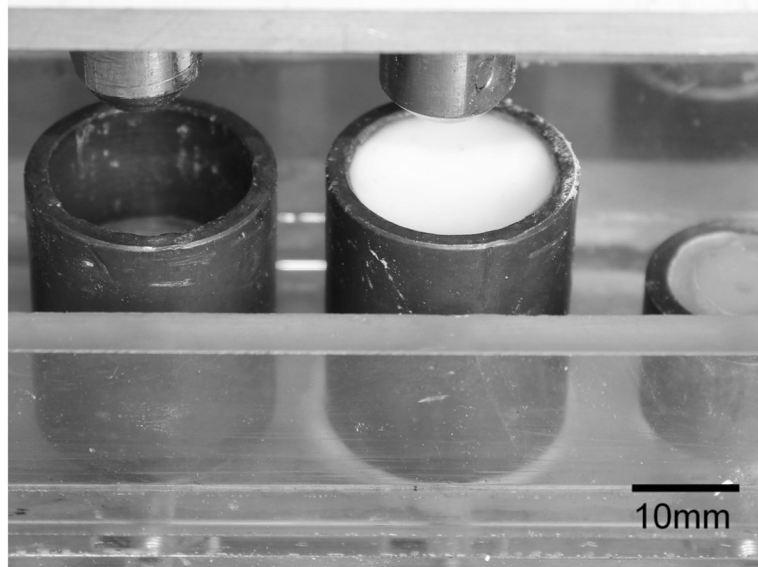


Figure 2. Brass wells of medium particles suspended over composite specimens

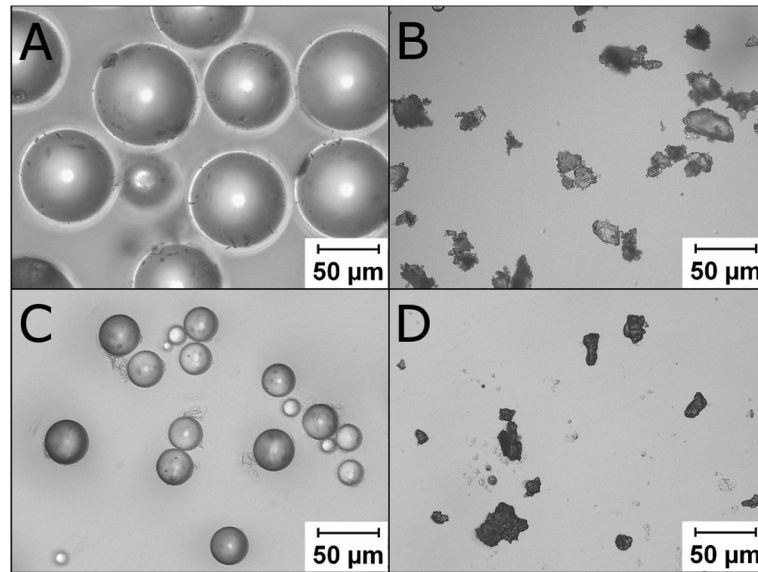


Figure 3. Micrographs of 4 medium particles (A) glass, (B) millet, (C) PMMA, (D) poppy

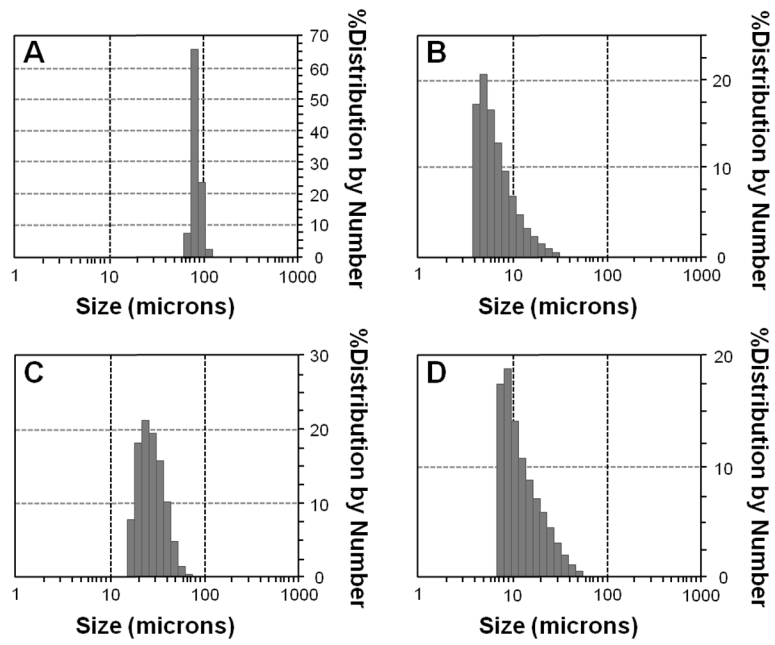


Figure 4. Particle size (by number) of 4 medium particles (A) glass, (B) millet, (C) PMMA, (D) poppy

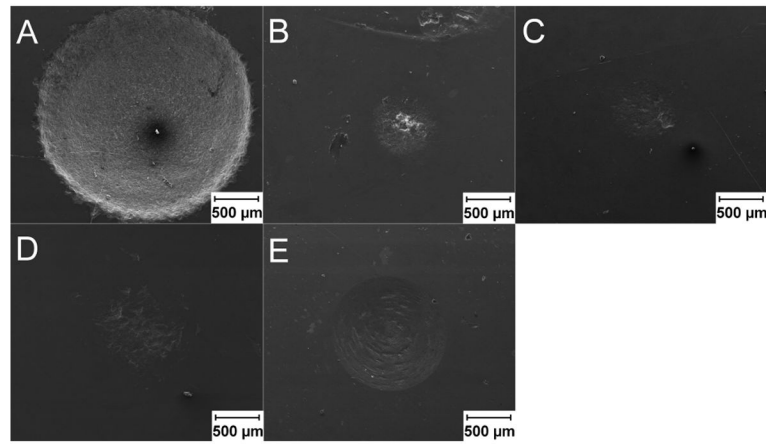


Figure 5. SEM images of worn surfaces of Filtek Supreme using different medium particles (A) glass, (B) millet, (C) PMMA, (D) poppy, (E) water

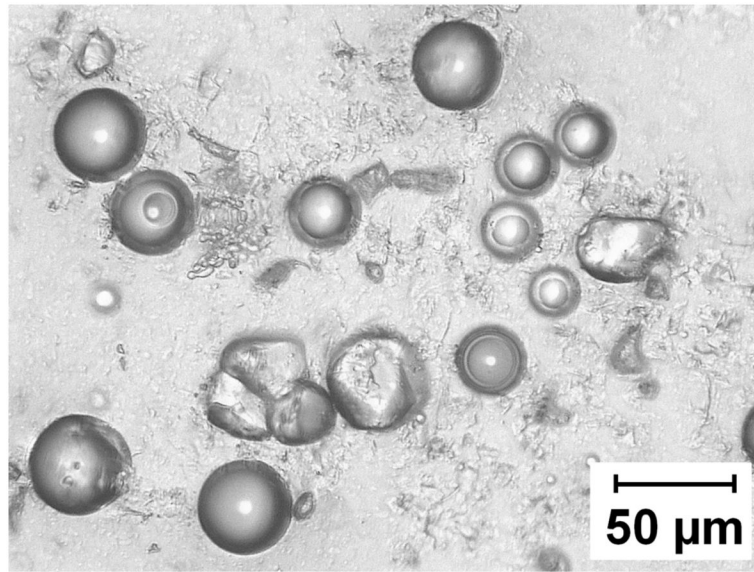


Figure 6. Micrograph of PMMA particles that have been roughened after wear testing

Table 1

Materials used in this study

Material	Classification	Lot No.	Matrix	Filler	Total filler content
Estelite Flow Quick	Flowable Microhybrid	UE 401236	Bis-EMA, TEGDMA, 1-6bis(methacryleth yloxy carbonyl)amino)trimethyl hexane	Silica/titania and silica/zirconia particles: 0.04-0.6µm	71% (wt) 53% (vol)
Esthet X	Micromatrix	060329	Urethane modified bis-GMA	Bariumalumino fluoroborosilicate glass: 0.02-2.5µm Silica: 10-20nm	77% (wt) 60% (vol)
Filetek Supreme Plus	Nano-filled	20060606	Bis-EMA6, UDMA, Bis-GMA, TEGDMA	Silica: 5-20nm nanoparticle Zirconia/silica: 0.6-1.4µm nanocluster	78.5% (wt) 57.7% (vol)
Z100	Hybrid	6KM	Bis-GMA, TEGDMA, 2-benzotriazolylmethylphenol	Zirconia/silica: 0.01-3.5µm	85% (wt) 66% (vol)

Bis-EMA = Bisphenol A polyethoxy methacrylate, TEGDMA = Triethylene glycol dimethacrylate, BisEMA6 = Bisphenol A polyethylene glycol diether dimethacrylate, UDMA = Diurethane dimethacrylate, Bis-GMA = Bisphenol A diglycidyl ether dimethacrylate

Table 2
Nano-hardness of composite materials used in this study

Material	Nano-hardness (GPa)
Estelite Flow Quick	0.303 (0.062)
Esthet X	0.657 (0.076)
Filtek Supreme Plus	0.873 (0.107)
Z100	1.101 (0.256)

Table 3
Nanohardness, form factor and particle size of medium particles

Material	Nano-Hardness (GPa)	Form Factor	Mean Particle Size (μm)
Glass	1.310 (0.150) [0.11]	1 (0) [0]	88.35 (8.24) [0.09]
Millet	0.279 (0.170) [0.61]	0.68 (0.20) [0.29]	8.07 (4.05) [0.50]
PMMA	0.279 (0.095) [0.34]	1 (0) [0]	28.95 (8.74) [0.30]
Poppy	0.226 (0.146) [0.65]	0.70 (0.30) [0.43]	14.08 (7.20) [0.50]

*Values displayed in [] represent COV

Table 4
Volumetric wear of composites using 4 medium particles and water

Material	Volumetric wear $\times 10^{-3}$ (mm ³) reported as mean (SD) [COV]			
	Estelite Flow	Esthet X	Filtek Supreme	Z100
Glass	868.88(72.94) ^{a C} [0.08]	1183.12(145.31) ^{b C} [0.12]	1343.51(140.02) ^{b,c D} [0.10]	1359.02(81.87) ^{c E} [0.06]
Millet	30.96 (57.82) ^{a A} [1.87]	14.51(32.68) ^{a A} [2.25]	56.86(49.32) ^{a B,C} [0.87]	1.94(2.90) ^{a B} [1.50]
PMMA	1.86(1.06) ^{b A} [0.57]	3.74(1.95) ^{c A} [0.52]	1.86(0.93) ^{b A} [0.50]	0.37(0.07) ^{a A} [0.19]
Poppy	2.64(3.21) ^{a A} [1.21]	10.30(8.28) ^{b A} [0.80]	17.34(29.42) ^{b A,B} [1.70]	17.88(16.79) ^{b C} [0.94]
Water	94.75(11.07) ^{c B} [0.11]	80.88(9.00) ^{b,c B} [0.11]	46.88(6.51) ^{a C} [0.14]	70.00(22.73) ^{b D} [0.32]

*Values displayed in [] represent COV

#Similar lowercase superscripts represent statistically similar groups in each row and similar uppercase superscripts represent statistically similar groups in each column