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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 1.2 Hz with 70 N 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, and poppy, respectively): 1.310(0.150), 0.279(0.170), 0.279(0.095), and 0.226(0.146). Average particle sizes (μm) are (glass, millet, PMMA, and 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.

1. 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

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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 hydroxyapetite, 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].

2. Materials and methods

2.1. Media particle preparation

Millet seed was prepared, as described by Nihei et al. [15], by grinding 50 g of seeds in a rotating blade grinder for 5 s. Poppy seed was prepared as described by Condon and Ferracane [7] by grinding 3 g 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.

2.2. Nano-hardness measurement

The medium particles were embedded in a 95% methyl methacrylate/5% *n*-butyl 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 h. 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). Indentions were depth controlled to 0.5 μm and performed with a diamond Berkovitch pyramid-shaped stylus (diameter = 40 nm). A 4 × 4 grid of indents (5 μm 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 (1 cm diameter × 4 mm) and light polymerized at 2 mm increments with a Coltolux LED curing light (Coltene/Whaledent, Cuyahoga Falls, OH, USA) (583 mW/cm^2). They were then polished using 600 and 1200 grit silicon carbon paper followed by 0.5 μm alumina slurry on a polishing wheel (Metallurgical polisher, Buehler Ltd., Evanston, IL, USA) at 80 rotations/s and 20 N of force. Nano-hardness of the composites was determined by creating a 4 × 4 grid of indents (5 μm spacing between indents) at two locations on the composite surface. The same testing parameters were used as described above.

2.3. Particle size measurement

The medium particles were mixed with distilled water in a 3:1 ratio. A 3 mL 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 24 nm and 2800 μm. Three measurements were taken of each sample, and media were sonicated for 2 min between measurements to prevent agglomeration.

2.4. Particle imaging and shape measurement

Particles were randomly dispersed on a glass slide. The particles were imaged with 1000× 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: $FF = 4\pi A/p^2$ [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.