Summary
This application note examines microbead-containing toothpaste and its proposed effects on simulated tooth enamel. Testing revealed wear on enamel material in brushing tests with both the conventional and the microbead toothpaste samples whereas the control sample (brushing without toothpaste) did not exhibit any signs of wear. It is known, however, that all toothpastes contain abrasives for cleaning surfaces. Minor surface scratching was seen on the conventional toothpaste sample, while cracking and subsurface chipping was seen on the microbead toothpaste sample. This data shows that microbead toothpaste is more abrasive and damaging to tooth enamel than conventional toothpaste.

Background

Many products, such as toothpaste and exfoliating facewash, contain plastic microparticles referred to as microbeads or microplastics. Within the last year, a great deal of attention has been focused on the drawbacks of microplastics in personal care products. Numerous concerns, from environmental to health issues, have been raised and the U.S. House of Representatives has approved a bill to phase out microplastics in personal care products beginning in 2017.1

Microbeads do not disintegrate or degrade, and many surpass filtration systems in water treatment centers. Studies have shown waterways and aquatic life riddled with microplastics while dentists are seeing an increasing number trapped in the gums of patients.2

Dental professionals emphasize the importance of keeping the gums and enamel healthy, and often urge us to rid ourselves of bad habits such as the consumption of acidic drinks. The sulcus, an area between the tooth and gum tissue, is particularly vulnerable. If microbeads become trapped in the sulcus, bacteria form, contributing to gingivitis and periodontal disease if left untreated. Abrasiveness of toothpaste is also of concern, since enamel is a tissue that does not regenerate.

Relative dentin abrasivity (RDA) is a method that quantifies the erosive effect of abrasives on the dentin, the inner layer of the tooth protected by enamel. During RDA testing, the enamel is completely stripped from the tooth exposing the dentin layer. Next, a slurry of toothpaste and water is used on radioactively marked tissue to quantify abrasiveness. According to ISO 11609, toothpastes with a RDA fewer than 250 are considered safe for lifetime use.3

Relative dentin abrasion method of measurement of dentifrice abrasiveness toward human teeth was described in 1958 by researchers R. J. Grabenstetter, R. W. Broge, F. L. Jackson, and A. W. Radike in their article The Measurement Of The Abrasion Of Human Teeth By Dentifrice Abrasives: A Test Utilizing Radioactive Teeth.4

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Both enamel and dentin tissues are made primarily of hydroxyapatite, a crystalline calcium phosphate\textsuperscript{5}. The enamel is composed of approximately 88\% mineral (hydroxyapatite), 2\% organic, and 10\% water whereas the dentin is composed of approximately 50\% mineral, 25\% organic, and 25\% water\textsuperscript{6}. Therefore, hydroxyapatite discs are used in this study to simulate enamel material.

**Samples and Materials**

For a simulated tooth enamel surface, 3D Biotek hydroxyapatite discs (approximate 5mm diameter, 1.6mm thickness) were used. Two toothpastes were used: Crest 3D White fluoride anticavity, arctic fresh icy cool mint toothpaste (micro-bead containing toothpaste); and Colgate Cavity Protection toothpaste, regular flavor (conventional). A new Colgate extra clean toothbrush (full head, soft) was used for each toothpaste type, as well as the control, which had the same brushing action but without any toothpaste slurry.

**Procedure and Results**

*Abrasiveness Testing*

Abrasiveness testing was performed using a custom testing apparatus (see Figure 2 below). A mold was constructed using silicones and allowed 3 separate testing reservoirs to be tested simultaneously. In the center of each reservoir, a 5mm diameter hydroxyapatite disc was held in place and submerged in water (control). A slurry of traditional Colgate toothpaste and a slurry of Crest 3D White toothpaste were added to two of the discs. The third disc was used as a control, and had no toothpaste slurry. A motor was used to power the toothbrush arm over the samples at a rate of approximately 60 rpm. A 250g polyethylene cylinder was used to add pressure, simulating real-time brushing. Based on recommended brush times, it was determined that 1 hour of simulated brushing was equivalent to approximately 1 month of real-time brushing. Each hour, the water/slurry levels in the reservoirs were checked and replenished as needed. Data was collected at 3 month, 6 month, 12 month, and 24 month equivalent time points on the Olympus STM6 microscope at 50X total magnification. Images of the enamel surfaces at the initial and final time points were collected on a Zeiss scanning electron microscope.

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\textsuperscript{6} http://pocketdentistry.com/4-fundamental-concepts-of-enamel-and-dentin-adhesion/
Results

Optical photographs of each disc were taken on the Olympus STM6 microscope using green fluorescent light to illustrate surface features. Blue arrows are used to show reference features on each image. Figure 3 and Figure 4 below show no visible differences between the initial and one year time points in the control (water) sample. Figure 5 and Figure 6 below show no visible differences between initial and one year time points in the conventional toothpaste sample. Finally, Figure 7 and Figure 8 illustrate differences between initial and one year time points in the microbead toothpaste. The blue arrows in Figure 7 and Figure 8 indicate a surface change indicated by darkening of the reference feature. A visual difference in abrasiveness is noted between the conventional and microbead toothpastes showing that microbeads are more abrasive to the enamel surface.

Scanning electron micrograph images for each sample were taken at initial and final (two year) time points. Figure 9 and Figure 10 show no new visible surface features between initial and final time points in the control samples. Figure 11 and Figure 12 illustrate the differences found between the initial and two year time points in the conventional toothpaste sample. Figure 12 shows visible surface scratching after two years of simulated brush time using conventional toothpaste. Figure 13, Figure 14, and Figure 15 also exhibit visible surface differences between the initial and two year time points for the microbead containing toothpaste. Figure 14 shows deeper subsurface chipping while Figure 15 shows cracking in the sample. All toothpastes are known to contain abrasives for cleaning purposes; therefore it is not surprising that small surface scratches are seen on the enamel of the conventional toothpaste samples. However, the microbead containing toothpaste sample has a higher degree of surface damage including cracking and subsurface chipping, suggesting that microplastics significantly increase the amount of damage to the enamel surface.

Figure 3: Micrograph of enamel surface of control sample, initial time point. Blue arrows indicate reference features.
Figure 4: Micrograph of enamel surface of control sample, one year time point. Blue arrows indicate reference features.

Figure 5: Micrograph of enamel surface of conventional toothpaste sample, initial time point. Blue arrows indicate reference features.
Figure 6: Micrograph of enamel surface of conventional toothpaste sample, one year time point. Blue arrows indicate reference features.

Figure 7: Micrograph of enamel surface of microbead toothpaste sample, initial time point. Blue arrows indicate reference features.
Figure 8: Micrograph of enamel surface of microbead toothpaste sample, one year time point. Blue arrows indicate reference features.

Figure 9: SEM micrograph of enamel surface of control sample, initial time point.
Figure 10: SEM micrograph of enamel surface of control sample, two year time point.

Figure 11: SEM micrograph of enamel surface of conventional toothpaste sample, initial time point.
Figure 12: SEM micrograph of enamel surface of conventional toothpaste sample, two year time point.

Figure 13: SEM micrograph of enamel surface of microbead toothpaste sample, initial time point.
Figure 14: SEM micrograph of enamel surface of microbead toothpaste sample, two year time point.

Figure 15: SEM micrograph of enamel surface of microbead toothpaste sample, two year time point.

Characterization of Microbeads

A toothpaste slurry containing a two inch strip of Crest 3D White toothpaste and tap water was filtered using simple vacuum filtration through a 25 µm filter and allowed to dry (see Figure 16). The microbeads were tested on a Varian FTIR using an ATR attachment. The spectrum obtained was run through Bio-Rad KnowItAll software, and it was found that the microbeads were indeed composed of polyethylene (see Figure 17). The OMX #1 A-C polyethylene had a hit quality index (HQI) of 888.7 and a percent fit of 88.7%, which is considered a very strong match.

In addition, a scanning electron micrograph of the polyethylene microbeads was taken (Figure 18). The microbeads are between 200 and 300µm and are rough and uneven in shape. These qualities suggest that the polyethylene used in Crest 3D White toothpaste is milled rather than formed by suspension.
polymerization. Since the microbeads are milled, they have sharper edges which may contribute to more significant abrasion than microbeads formed by suspension polymerization.

Figure 16: Filter paper containing microbeads from Crest 3D white toothpaste

Figure 17: FTIR spectra of blue microbeads from Crest 3D White toothpaste (bottom) with OMX #1, A-C polyethylene (top)
Figure 18: Scanning electron micrograph of polyethylene beads from Crest 3D White toothpaste