Simulated Toothbrushing Wear of Different Composite Resin Restorative Materials

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***Abstract* - This study evaluated surface roughness after simulated toothbrushing of five commercial composite resin restorative materials: one microhybrid: Denfil; one Ormocer: Admira; two nanohybrids: Grandio and Ceram X mono; and one Giomer: Beautifil II. Cylindrical specimens of five composite resin (n=12 for each material) were used. All samples were brushed for 200 min with dentifrice-water slurry in a simulated tooth-brushing machine. This period is clinically equal to two years age of toothbrushing. Before and after simulated toothbrushing wear para-meters for surface roughness (Ra, µm) were determined by a surface profilometer respectively. The data was statistically analyzed by one way analysis of variance (ANOVA) and Tukey's post hoc test to evaluate the difference among the tested materials and Paired Sample T test to evaluate the surface roughness changes after simulated toothbrushing. And then, comparing the surface roughness among the tested materials: Denfil, Admira, Grandio, Ceram X mono and Beautiful II. SEM observation was done to evaluate the changes in surface topography of all tested composite resin materials before and after simulated toothbrushing. The mean percentages for surface roughness, initial surface roughness means ranged from 0.2081 (0.0672), 0.1571 (0.0294), 0.1977 (0.0642), 0.1977 (0.0679), and 0.1822 (0.0641) for Denfil, Admira, Grandio, CeramX mono, and Beautifil II respectively to 0.3072 (0.0957), 0.2331 (0.0489), 0.2315 (0.0705), 0.2252 (0.0880), and 0.1995 (0.0605) after testing. After simulated toothbrushing, statistically significant change in weight loss and surface roughness was detected in all tested materials (*p*<0.01). The results of this study suggested that some important general relationships exist between the compositions, micro-structures and mechanical properties of tested composite resins. The analysis of differences before and after the simulated toothbrushing may provide some further basis for a rational choice of the most appropriate composite resin material from wearing aspect.**

Keywords; toothbrushing, surface roughness, composite resin

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Introduction

Research in the aesthetic dentistry seeks to meet to the demands of the clinicians, who desire restorative materials with easier handling characteristics and superior physical, mechanical and aesthetic properties. However, it is known that achieving all these characteristics in one material constitutes a difficult task (Chimello *et al*., 2001). In that, the most important property is the ability to withstand wear, as any loss of substance could result in altering the anatomic shape and affecting the performance of restoration. Nowadays, composite resins are so far the most esthetic restorative material applied in direct restorations. These materials offer the patients with minimally invasive, highly aesthetic in performance. However, the aesthetic property of these materials is unpredictable due to their discolouration or loss of luster, owing to surface wear.

Despite improvements in mechanical properties and wear resistance in composite materials, wear is still a cause of clinical failure, particularly in high stress areas such as molar restoration (cited in Sarett, 2000). Decreasing wear resistance created the surface roughness of restorations and complicated as plaque accumulation, loss of surface gloss and further attack of caries development. Therefore, adequate wear resistance is the most important requirement for posterior restoration. However, numerous studies have been carried out to investigate wear of composite resin restorations and most of them showed that wear is inevitable. During maintaining oral health and restorations, dental tissues and dental materials are subjected to tooth brush/ toothpaste abrasion, but an impact of toothbrushing on appearance of composite resins may occur (cited in Strassler, 2010). This may cause wear on dental tissues and dental materials during removal of surface deposits especially in the cervical third area of facial surfaces of the teeth. This effect leads to changes in the surface condition of any composite material.

As many researchers reported conflicting papers on the ability to predict wear from the physical and mechanical properties of composite resins, actual testing and measurement are required. However, clinical tests (*in vivo*) are not easily performed, as there are problems associated with the type of trial tests. Simulation testing should achieve in the shortest possible time for the test to be a useful alternative to a protracted clinical trial (Mandikos *et al*., 2001). Up to now, a more reliable way of studying wear resistance in vitro is to use a toothbrush machine with dentifrices and prophylaxis materials. It simply simulates the wear mechanism that occurs during toothbrushing. The surface roughness produced by a tooth brushing abrasion test reflects the abrasive resistance of the resin materials.

The type of composite material is especially important from a clinical standpoint, because the type and composition of the current composites introduced as restorative materials differ widely. Thus, many researchers reported that evaluation of the mechanical properties of restorative materials is necessary to ascertain their indications and limitations. As a matter of fact, wear of tooth coloured restorative materials is still concerning. Controversial results were determined associated with various degree of wear resistance of restorative materials to different degree of abrasivity and various testing methods. Hence, it is necessary, to understand the wear resistance of different composite materials. In this study, it was taken into consideration to evaluate the surface roughness of different composite resin restorative materials with respect to the esthetics and function of restorations which are important for longevity of restorations.

Materials and Methods

The wear of five commercial composite resin restorative materials were compared after simulated toothbrushing: one microhybrid: Denfil; one Ormocer: Admira; two nanohybrids: Grandio and Ceram X mono; and one Giomer: Beautifil

II. List of these materials used, manufacturers, composition, and batch number are shown in Table 1.

Table 1. Details of tested resin materials

Trade name	Manufacturer	Lot number	Main composition	Average Filler particle size	Filler content wt% / vol%
DenFil	Vericom, Korea	DF 2417922	Microhybrid Bis GMA, TEGDMA	≤1.0 µm	80% wt
Admira	VOCO,G mbH Germany	1002512	Ormocer based microfilled 3-dimensionally linked inorganic – organic copolymer (ORMOCERS) Additive aliphatic aromatic dimethacrylate	0.7 µm	56% vol
Grandio	VOCO,G mbH, Germany	1047274	Nanohybrid Matrix: Bis-GMA, UDMA,TEGDMA, dimethacrylate	0.1 – 2.5 µm$^{\beta}$ 20 -50 nm$^{\theta}$	71.4 % vol 87% wt
Ceram® X mono	Dentsply Asia	13030000 81	Nano-ceramic Methacrylate mod-ified polysiloxane dimethacrylate resin	1.1– 1.5 µm	57% vol 76% wt
Beautifil II	SHOFU, Japan	031135	S-PRG filler	10-20 nm	83.3 %wt

Twelve cylindrical specimens were made of each material using a stainless steel mould (2 mm thick and 9 mm inner diameter). Each material was inserted directly into a mould with a plastic instrument. A microscope glass slide (Mariefeld, Germany) interposed with a cellulose matrix strip (PN 1148, SHOFU, Japan) was placed on the composite resin to avoid air inhibition layer (oxidizing layer) and to obtain a smooth surface with the polish of natural resin. Finger pressure was used for 30 seconds, to remove the excess and obtain a flat surface. After this time, the pressure was removed. The specimen was polymerized in accordance with the manufacturer's recommendations, i.e. 40 seconds, covering the total area with a high intensity visible light (LED) source (Guilin Woodpecker medical instrument Co. Ltd, Guangxi, China) with an output of 800 mW/cm2 at all times keeping the tip as close as possible to the resin surface and perpendicular to the restoration surface (by touching to the surface of glass slab at all times). The output from the curing light was periodically monitored with a curing radiometer (DigiRate radiometer, LM-100, Monitex, Taiwan). Polymerizing the composite materials against a glass surface is a method commonly used by researchers to produce a standardized surface finish for testing (Tanoue *et al*., 2000). The chosen technique excluded air from the composite surface and thereby minimized oxygen absorption to produce an oxygen-inhibition layer. The degree of attenuation of the high–intensity curing light due to the borosilicate microscope slide (1 mm thick) was judged to be negligible. To reduce variability, the same shade of composite materials (shade–A3) was used and operated by the same operator. The specimens were examined for obvious voids, bubbles, and surface imperfections with handy magnifying glass (magnification × 3). The specimens were labeled on the bottom.

The samples were stored in distilled water at 37 degree centigrade for 24 hours to allow completion of polymerization reaction. Moreover, they would get hygroscopic expansion and would not absorb dentifrice slurry during tooth-brushing test.

Thereafter, the specimens were polished with wet 800- grit, followed by 1000-, 1200- , 1500- and 2000 grit silicon carbide waterproof electro coated abrasive papers in a polishing machine under running water to obtain a plane, smooth and glossy surface. Each grit was ground for 10 sec. The rationale for the use of

2000 grit silicon carbide paper is to produce an optimally polished surface and to have a comparable initial roughness which allowed us to better assess the materials' roughness as well as to remove the oxidizing layer. After finishing and polishing, specimens were rinsed with running tap water. Then ultrasonic cleaning of the polished specimens was performed for 3 minutes in water to remove any surface debris. Afterwards, each specimen was dried with absorbent paper to remove excess water. And then each specimen was dried with gentle air. To ensure the dryness, the specimen was placed in air for 24 hours. All the procedures were performed under ambient light and temperature.

The alteration in surface topography of specimens was initially measured with surface roughness tester (SRT-6200, Lanetech Co. Ltd, Beijing, China) to verify the roughness (Ra) values of the surfaces before tooth brushing. To measure the roughness profile value, the diamond stylus was moved across the surface under a constant load. The instrument was calibrated using a standard reference specimen. Five readings were made on each surface using a stylus tip (2 µm in diameter).

The surface was profiled at five different locations for each specimen. These measurements were one in centre area and four different directions through the centre of the. For every reading made, the mean roughness value (Ra, µm) was represented by the arithmetic mean between the peaks and valleys registered. After the stylus of the profilometer had scanned, a stretch of 2.85 mm in length, with a cut-off of 0.25 µm to maximize the filtering and undulation on the surface. Initial roughness data was expressed as R1. In order to study the appearance of the polished and finished surface of the specimen as a baseline, a scanning electron microscope was used. Scanning electron microscopy (SEM) has been largely used in dentistry to study surface characteristics of different materials. Only two samples for each group which Ra values tested by surface roughness tester are closest to mean value were selected.

The surface of the specimens was sputter-coated with platinum and then observed using a scanning electron microscope (JEOL JSM – 5610 LV, Japan) with a magnification of 550× and 5500×. To evaluate the same specimen after toothbrushing, the palladium sputter was removed with alcohol before submitting the specimen to the toothbrushing device.

The samples were identified and subjected to the mechanical tooth brushing test. Each specimen was fixed on a specimen holder attached to a toothbrush abrasion testing machine. In tooth brushing simulating machine, Oral B electric tooth brush (Oral B: Advance Power 400, powered by BRAUN, Germany) was fixed to the apparatus and kept alongside the samples, using slurry of Colgate® Total 12 Clean Mint dentifrice with deionized water in a ratio of 1:2 by weight, according to the ISO specification (cited in Mondelli, 2005).The dentifrice was placed between the brush and the surface of the material to maintain constant supply of abrasive during brushing. The water level of the slurry was kept high enough to ensure that the brushing surface was under the dissolved dentifrice. The dentifrice slurry was regularly changed after 100 minutes of brushing time which is clinically equal to one year age. The slurry pH was checked during the test (between 7.0 and 7.4). Simulated toothbrushing was performed with rotation and oscillation action (oscillating at 7,200/min). Bristles are flexisoft and colour fade was indicated to replace brushhead. In the present study, each brushhead was used for only one specimen. The test was made under a load of 350 grams, to simulate the force of tooth brushing procedures. Each specimen was brushed for 200 minutes (3 hours and 20 minutes). According to Heintze's work (2010), this period is clinically equal to two years age of toothbrushing. Following the test, the specimens were removed. Specimens were rinsed with running tap water to remove surface debris and ultrasonically cleaned with water for 3 minutes. And then each specimen was dried with gentle air. To ensure the dryness, the specimen was placed in air for 24 hours. Final roughness measurements were made according to initial measurement protocols. The measured final roughness data was expressed as R2.

The roughness alteration (changes in surface topography) was observed by the difference between baseline and final reading means. In order to study the changes in surface topography of different composite resins after toothbrushing, a scanning electron microscope was used. For each group, Ra values which were nearest to the mean values were selected. The wear resistance was evaluated through the averages of surface roughness. This study was approved by Research and Ethical Committee of University of Dental Medicine, Yangon.

Statistical Analysis

The data was statistically analyzed by one way analysis of variance (ANOVA) and Tukey's post hoc test to evaluate the difference among the tested materials. Paired Sample T test was used to evaluate the surface roughness changes after simulated toothbrushing. The results were analyzed by calculating the mean and standard deviations for each experimental group.

Results

The results of average surface roughness before and after simulated toothbrushing can be observed in Figure 1.

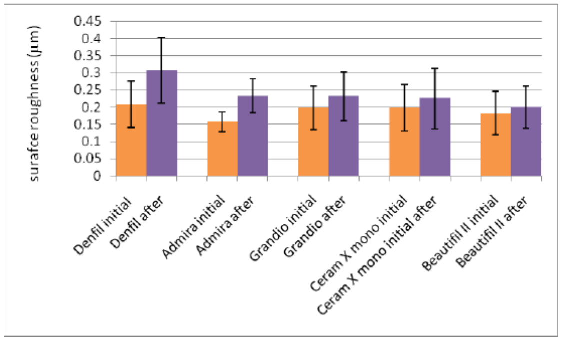

Figure 1. Bar chart showing surface roughness of different composite resins before and after simulated toothbrushing (Mean±SD) (Error bar denote standard deviation)

It shows graphically comparison of average surface roughness alteration of different composite groups after simulated toothbrushing. All tested composite materials demonstrated more surface roughness values after simulated toothbrushing wear. Denfil and Admira groups showed greater surface roughness alteration than Grandio, Ceram X mono, and Beautiful II specimens (p<0.01).There were no statistical differences between Grandio, Ceram X mono, and Beautiful II (p>0.05). However, Beautifil II presented the least surface roughness alteration result, and Denfil showed the largest degree of surface roughness alteration without statistical differences with Admira.

Scanning Electron Microscope (SEM) Observation

The surface topography of all tested

composite resin materials before and after simulated toothbrushing wear were compared in Figure – 2.A, B, C, D, E. The SEM microphotographs in Figure 3 illustrate the surface texture of tested resin composites based on surfaces pre-ground with 800, 1000, 1200, 1500, and 2000 – grit SiC paper respectively (done as finishing procedure before simulated toothbrushing) and surface wear after simulated toothbrushing.

Generally, all composite groups tested in this study exhibited rougher surface texture after toothbrushing. When observing the images of SEM before toothbrushing, Denfil and Admira groups revealed the grinding scratches on the surface owing to finishing procedures. Moreover, some area of surface of Admira presented grooves and facets. Remaining three groups showed no scratches and mostly smoother surfaces than previous two resins. However, exposure of inorganic filler particles which distributed homogenously in different shapes and sizes was detected. Among the tested materials, fillers of Ceram X mono was the smallest in size and homogenously distributed, although Grandio and Beautifil II distributed irregular and larger filler particles and among them nano sized particles were filled. In addition, some areas of SEM images showed voids appearance. When observing the images of tested materials after simulated toothbrushing, a combination of filler exposure, loss of inorganic particles and resin matrix wear were detected. When analyzing the size of holes and craters, there was no broader than filler particle sizes. Hence, it may be due to exfoliation of filler particles. However, in case of Denfil and Beautifil II, there may be occurred a few larger holes, it may be due to dislodging of filler particle and resin matrix couple clusters.

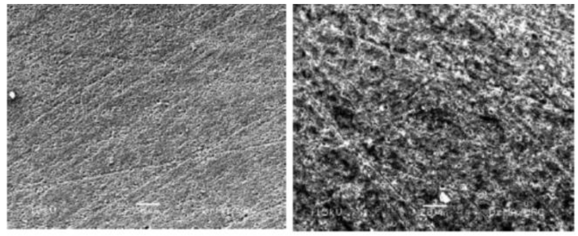

Figure 2A. SEM microphotographs of Denfil composite before and after simulated toothbrushing wear (550×)

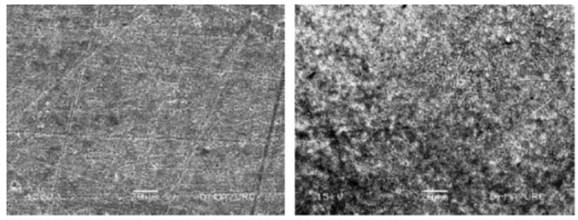

Figure 2B. SEM microphotographs of Admira composite before and after simulated toothbrushing wear (550×)

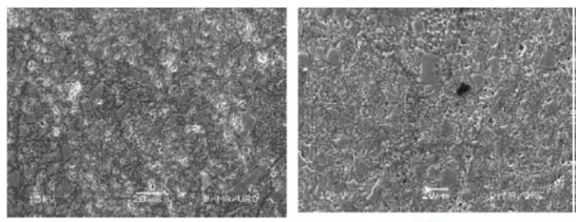

Figure 2C. SEM microphotographs of Grandio composite before and after simulated toothbrushing wear (550×)

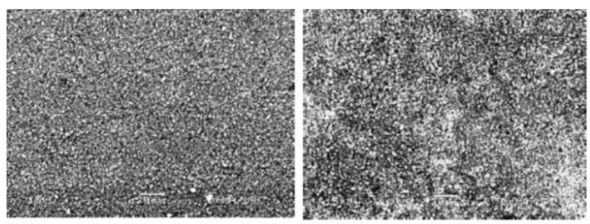

Figure 2D. SEM microphotographs of Ceram X mono composite before and after simulated toothbrushing wear (550×)

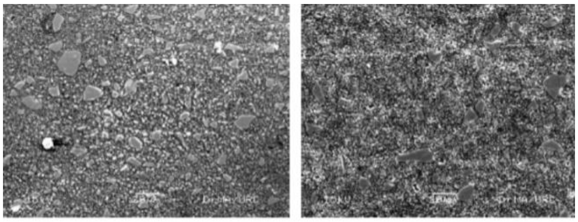

Figure 2E. SEM microphotographs of Beautifil II composite before and after simulated toothbrushing wear (550×)

Discussion

The ultimate goal of continuous material research and development is to enhance the practice of dentistry. The present study assessed surface roughness of different composite resin restorative materials after simulated toothbrushing.

The simulated toothbrushing abrasion with dentifrice slurry caused significant surface roughening for all tested resin materials. Moraes *et al.*, (2007) reported that the roughening effect could be rather significant, and the materials with finer particles showed lower roughening. Similar findings of the present study agreed with their results except for Beautifil II (Giomer) material.

Concerning with Beautifil II composite (Giomer), while analyzing with profilometer, its surface roughness was the least among the tested groups, although there were no statistically differences between Grandio, Ceram X mono and Beautifil II (p>0.05).

Composite resins have a mixed structure consisting of soft matrix resin and hard filler particles. Unfortunately, there usually is a problem during the toothbrushing. Since the resin matrix and inorganic filler differ in hardness, surface of the composite resins are not abraded uniformly. After toothbrushing, soft matrix resin was firstly worn and the dislodgment of filler particles was caused by long-term use (Tanoue *et al.*, 2000).

The SEM finding of the present study supports this phenomenon. Simulated toothbrushing created scratches on the surfaces of denfil composite (Figure 2A) and plucked the particles away, while a uniform smooth surface could be generally obtained in remaining groups. However, the roughness values were not the same for these groups.

While studying the SEM micro-photographs of tested composites after simulated toothbrushing, a seemingly rather smooth surface was achieved for Admira (Figure 2B) at 550× magnification, but examination at 5500× magnification revealed a less homogenously abraded surface and the exfoliated small filler particles and created the crater like area. Therefore, after the simulated toothbrushing with dentifrice slurry, the surface texture of all tested resin materials were apparently abraded under scanning electron microscopic examination. With the present outcomes, this study agreed with the results of Strassler *et al*., (1988) in which reported that toothpastes can affect composite smoothness. Furthermore, Ergucu and Turkun (2007) observed that profilometric measurements were strongly confirmed by SEM analysis.

While observing the SEM micrographs of the tested materials after the simulated toothbrushing, nanohybrid resins, (Grandio-Figure 2C and Ceram X mono-Figure 2D) revealed lesser values of surface roughness than that of microhybrid resin (Denfil). It was agreed with the work of Oliveira *et al.*, (2012) where they reported that the qualitative analysis of the SEM micrographs of the resins, after the abrasion test, showed more polished surface of the nanofilled and the nanohybrid resins than the microhybrid resins. In addition, they proved that Admira (Ormocer based composite) was significantly rougher than the nanofilled composites.

In this aspect, the most likely cause of the improved abrasion resistance of the nanohybrids in comparison with microhybrid counterpart lies in differences between filler technologies of these materials. Filler particles should be situated as close together as possible in order to protect the resin matrix from abrasion. The application of nanotechnology to composite research is of great benefit. Due to reduced dimension of the particles and wider distribution, an increased filler load can be achieved, which results in reducing polymerization shrinkage and increasing mechanical properties (Terry, 2004).

Manhart *et al.*, (2000) and Tanoue *et al.*, (2000) mentioned that wear resistance of composite resins could be influenced not only by filler content and size but also by other factors such as matrix-filler interaction. In addition, five possible mechanisms for wear in composite resins: wear of the resin matrix, loss of fillers by failure of bonding to the matrix, loss of filler through shearing of exposed particles, loss of filler through cracking and failure of the matrix; and exposure of entrapped air bubbles (cited in Xie, 2000). The apparent contrast, particularly, between Admira and Grandio may be due to compositional differences in resin matrix and the filler, although they were produced by the same manufacturer. Differences in resin matrix composition may cause the variation in degree of conversion resulting in the different degree of wear. In general, resin matrix was composed of various amounts of Bis – GMA, Bis – EMA, UDMA, TEGDMA according to manufactures' formulations. Depending upon the percentage of TEGDMA, there may be changes in degree of conversion and plasticization (Filho *et al.*, 2008).

However, Soderholm *et al.*, (2001) stated that filler type and loading had little apparent effect on the wear observed. In the present study, Denfil group showed the worst value of surface roughness. It may be due to larger filler particle than other tested groups. The possible explanation relies on the fact that Denfil, even classified as a microhybrid composite, the filler size is ≤ 1 µm. These results are in agreement with data reported by Moraes *et al.*, (2007).

It is essential to determine the initial roughness to establish a parameter of comparison. Admira presented the smoothest surface. It may be due to the fact that it is an Ormocer based composite resin which is able to resist traditional finishing and polishing procedures. In addition, results of initial surface roughness of Grandio and Ceram X mono had no significant difference. This was somehow expected as both them are categorized as nanohybrids.

According to the present study, the surface roughness outcomes revealed variability of performance after simulated toothbrushing. In this study, Admira showed higher surface roughness values while Grandio had lower surface roughness after the simulated tooth brushing. Both composites were produced by the same manufacturer and composed of the same average particle size, 0.7 µm inorganic filler. However, the difference lies in fillers volume percentage (56 vol% and 71.4 vol% respectively). This causes the variation in inter-particle distance. If the distance is farther, resinous matrix can be abraded more leading to increase in the surface roughness. The largest roughness is responsible for an undesirable loss of esthetics of the restoration, due to the loss of surface gloss and biological disadvantages, causing dental plaque accumulation and increasing the risks of occurrence of caries and periodontal inflammation (Bollen, 1997).

In the present study, nano groups showed less surface wear changes than other groups except Giomer (Beautifil II). By understanding how particle size, distribution, and volume affect the mechanical properties of composite resins, the clinician can accurately diagnose and treat a patient's condition by selecting the appropriate composite resin restorative material. The assessment of the materials in the present study presented different outcomes for surface roughness changes, and it is clear that the mechanisms accounting for these phenomena are more complex than can be explained by filler component alone. Other factors should be also considered. Nevertheless, the conditions established here did not take into account the effects such as variations in pH, composition and temperature of saliva, food and beverages that can fluctuate in the oral cavity, the presence of plasticizing agents and mechanical fatigue such as sliding, abrasion, and the interaction of other stresses that could be encountered in the clinical situation.

It must be acknowledged that this study has several limitations. First, the *in vitro* nature of the present experimentation may limit, at least in part, its applicability to clinical practice. For example, present *in vitro* study correlates only to clinical situations in which there are accessible and relatively flat surface. Hence, the selection of the most appropriate material under these circumstances was recommended, the results obtained both from *in vivo* and *in vitro* should be evaluated together.

Conclusion

The findings in the present study showed that Ceram X mono and Beautifil II maintained comparatively smoother surface than that of other tested materials. But Beautifil II could not resist the wearing process. Therefore, Ceram X mono, one of nanohybrid, is the most reliable aesthetic restorative material that can resist toothbrushing wear comparatively within the limitation of present study.

The results of this study suggested that some important general relationships exist between the compositions, microstructures and mechanical properties of tested composite resins. Although intensive studies have been carried out throughout the decades to produce the most appropriate composite resin restorative materials, there are still considerable differences between the mechanical properties and wear resistance of tooth tissue and composite resins.

The analysis of differences in surface roughness before and after the simulated toothbrushing may provide some further basis for a rational choice of the most appropriate composite resin material.

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The authors declare there is no potential conflict of interest.

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