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An *in vitro* evaluation of wear and surface roughness of particulate filler composite resin after tooth brushing

ABDUL AZIZ A AL KHURAIF

Dental Health Department, College of Applied Medical Sciences, King Saud University, Riyadh, Saudi Arabia

Abstract

Objective. To evaluate the influence of tooth brushing on wear and surface roughness of four different particulate filler composite resins. ***Materials and methods.*** Six specimens (2 mm thick and 8 mm in diameter) of each tested material (Filtek Z250-Microhybrid, SpectrumTPH3-Submicron hybrid, Filtek Z350XT Nanofiller and Filtek P90- Microhybrid) were prepared according to the manufacturer's directions. A brushing sequence of 5000, 10 000 and 20 000 cycles was performed for all the samples. A non-contact profilometer was used to determine average surface roughness (Ra) and wear of the material assessed using an analytic electronic balance at baseline and each cycle interval. The data obtained were analyzed using one-way ANOVAs and post-hoc multiple comparison tests. Paired *t*-test was used for comparisons between cycle intervals for each material. Analyses with scanning electron microscopy (SEM) were also performed. ***Results.*** The resin composite Filtek P 90 presented an increase in percentage weight loss after final toothbrushing cycles over the rest of the materials. Brushing significantly increased roughness (Ra) for all composites. Filtek Z250, after brushing, was significantly rougher than the other resins followed by Filtek P 90, Spectrum TPH 3 and Filtek Z350 XT. However, SEM images indicated severe change in surface topography of 'sub-micron hybrid' specimen compared to each other after tooth brushing. ***Conclusions.*** Wear and surface roughness increased with each cycle interval for all the materials and one composite resin demonstrated a higher increase in surface roughness than the other three tested brands of composite resins. Not much difference was observed in the weight loss between tested samples.

Key Words: *composites, wear, surface roughness, tooth brush abrasion*

Introduction

Composite resins are mercury-free and esthetically attractive restorative materials that are widely used by clinicians. Composite resins vary by the filler type and content, which influences their handling characteristics and physical properties [1]. It is known that restorative materials undergo wear (in a manner similar to enamel and dentin) and the wear mode depends on the type of restorative material [2]. This may occur as a result of direct contact between the tooth and the restorative material during mastication, oral parafunctions, tooth brushing with abrasive particles and chemical effects caused by dietary factors [3].

Abrasion is an undesirable phenomenon, not only leading to an increase in surface roughness, but also resulting in the gradual removal of substance [4]. Restoration roughness increases the coefficient of friction and may increase the rate of wear [5]. Rough surfaces enhance accumulation of dental biofilm, residues and dyes, not only causing gingival irritation and risk of secondary caries, but also diminishing the gloss of the restoration, giving rise to discoloration and/or surface degradation [6,7]. Change of the surface properties relates to the wear of the polymer matrix and loosening of filler particles. Loosening of the fillers may occur due to leaching of the surface of filler particle or due to leaching of the silane promoted coupling interface. Profilometry is the most commonly used method to evaluate the surface quality in terms of surface roughness of composite materials [8].

Since composites consist of filler particles dispersed in a brittle polymer matrix []2, the polymer matrix and the extent of polymerization of the polymer matrix

Correspondence: Abdul Aziz A. Al Khuraif, Professor, Dental Health Department, College of Applied Medical Sciences, Dental Biomaterials Research Chair, King Saud University, PO Box 10219, Riyadh 11433, Saudi Arabia. Tel: +00966554777685. E-mail: aalkhuraif@ksu.edu.sa

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influence the wear characteristics of the composite. Traditionally, filler particles have been dispersed in dimethacrylate resin matrix which forms a brittle cross-linked matrix. A recently introduced silorane-based resin matrix is utilizing the anionic ring opening polymerization system which also results in a cross-linked matrix, but may have different wear resistance. Silorane was so named to indicate a hybrid compound of siloxane and oxirane functional moieties. While the siloxane determines the highly hydrophobic nature of the siloranes, the cycloaliphatic oxirane functional groups are responsible for lower shrinkage when compared to methacrylate-based composites. Oxiranes, which are cyclic ethers, polymerize by a cationic ring opening mechanism, while methacrylates polymerize via a free radical mechanism [9]. Therefore, it is hypothesized that the wear and surface roughness of restorations have a negative impact on the longevity of the restoration in an oral environment. The purpose of the study was to evaluate the wear and change in surface roughness of four commercial composites subjected to tooth brush–dentifrice abrasion at different cycle intervals using a tooth brush simulator.

Materials and methods

Four different particulate filler composite resins were selected: Filtek Z250-microhybrid, SpectrumTPH3-submicron hybrid, Filtek Z350XT-nanofiller and Filtek P90-microhybrid. The composition of the tested materials is shown in Table I. Five disc shaped specimens were made of each material using a mold (2 mm thick and 8 mm in diameter). Each material was inserted into the mold in one increment with an appropriate instrument. A glass slide was placed over the mold/resin under pressure, obtaining a flat surface, and the material was light-cured for 20 s each, as recommended by manufacturer's instructions, using a visible light-curing unit with 1200 mW/cm output (Elipar freelight 2, 3M ESPE, Seefeld, Germany). The samples were removed from the mold, stored in distilled water at 37°C for 7 days and, after this period, the surfaces opposite to the glass slide were finished and polished with Flexi snap kit (Edenta AG, Hauptstrasse, Switzerland) sequential disks from the coarsest to the finest granulation. Each specimen was weighed in an analytic electronic balance (Precisa EP 225 SM –DR, Precisa Gravimetrics AG, Switzerland) to determine its initial mass in grams (M1). Surface roughness was assessed using a non-contact profilometer (Bruker Contour GT, Tucson, AZ) for determining initial surface roughness (Ra_i). This surface profiling system utilizes Nanolens AFM module with fully automated turret and programmable X, Y and Z movements. This provides high resolution data of the surface. The simple vision 64 software transforms these high resolution data into accurate 3D images. Five random scans were carried out on the surface of each specimen and their mean corresponded to the initial surface roughness. Once the values for initial mass and roughness were obtained, the specimens were subjected to tooth brushing wear. For the wear test a tooth brushing simulator (Tooth brush simulator ZM 3, SD Mechatronik GMBH, Feldkirchen-Westerham, Germany) was used. The simulator was equipped with 12 independent slots to which 12 tooth brushes could be attached. A plexiglass pattern with the same dimension of the specimen was positioned inside the containers. Each specimen was fixed on the plexiglass using a drop of acrylic resin monomer on the surface of the plexiglass and the sample, pressing it against the plate. The container was filled with slurry of dentifrice (Colgate, Colgate Palmolive Arabia Ltd, Riyadh, Saudi Arabia) with particle size ranging from 4–12 μm and distilled water in a ratio of 1:1 by weight. A tooth brush with straight soft bristles (Oral B Classic, Gillette India ltd, Rajasthan, India) was used for the purpose. The slurry completely covered

Table I. Composition of materials used in this study*.

Material	Composition		
	Matrix	Filler	Filler size
Filtek Z Microhybrid	250 Bis-GMA, Bis-EMA, UDMA, TEGDMA	Silicon dioxide, Zirconium dioxide	0.01–3.5 μm
Spectrum TPH 3 Sub-micron hybrid composite	Bis-GMA, Bis-EMA, TEGDMA	Barium-Aluminium, Boro silicate, Silicon-di-oxide	0.04–5 μm
Filtek Z350 XT Nanofiller	Bis-GMA, Bis-EMA, UDMA, TEGDMA, PEGDMA	Silica, Zirconia	20–75 nm
Filtek P 90 Microhybrid	Silorane Resin	Quartz and yttrium fluoride	0.1–2 μm

*Product information as supplied by the manufacturers.
BIS-EMA, bisphenol A-polyethylene glycol diether dimethacrylate; BIS-GMA, bisphenol A-glycerolate dimethacrylate; PEGDM A, polyethylene glycol dimethacrylate; TEGDMA, tetraethylene glycol dimethacrylate; UDMA, urethane dimethacrylate.

Table II. Mean wear (grams) of tested composites after different tooth brushing cycles.

Material	Initial mass (Mi)	Mass after 5000 cycles (M5)	Mass after 10 000 cycles (M10)	Mass after 20 000 cycles (M20)	Percentage loss (ML)
Filtek Z 250	0.3729^{A}	0.3714^{B}	0.3710^{C}	0.3703^{C}	0.673^{a}
Spectrum TPH 3	0.3544^{A}	0.3541^{B}	0.3534^{C}	0.3526^{D}	0.510^{a}
Filtek Z 350 XT	0.3140^{A}	0.3135^{B}	0.3127^{C}	0.3119^{D}	0.675^{a}
Filtek P 90	0.3497^{A}	0.3488^{B}	0.3481^{B}	0.3472^{D}	0.706^{a}

Same upper case letter within a row indicates no difference (paired *t*-test, $p < 0.05$).
Same lower case letter within a column indicates no difference (Tukey's test, $p < 0.05$).
$n = 6$.

the samples and was monitored carefully for any refilling during the cycles. Tooth brushing was accomplished with a horizontal movement (forward and backward) under a load of 0.2 N and the stroke rate was 120 cycles/min with a stroke length of 38 mm. The tooth brushes were changed and slurry refilled every 2000 cycles. The test was repeated at three different tooth brushing cycle intervals (5000 cycles, 10 000 cycles and 20 000 cycles) apart from the baseline. After each test, the specimens were carefully removed from the plexiglass with the aid of a scalpel, rinsed in running water, dried and weighed, obtaining the mass (M5, M10, M20) and post-abrasion roughness (Ra5, Ra10, Ra20) as described for the initial mass and roughness test; The data obtained were analyzed using one-way ANOVAs and post-hoc multiple comparison test to determine whether there were significant differences. Paired *t*-test was used for comparisons between cycle intervals for each material. SEM micrographs of the representative surfaces of test samples before and after wear test were taken.

Results

The mean values of wear at baseline and after each toothbrushing cycle are shown in Table II. A statistically significant difference was found in the weight between baseline and after toothbrushing cycle ($p < 0.05$), except for Filtek P 90 at 10 000 cycles and Filtek Z 250 at 20 000 cycles, while no significant difference ($p = 0.26$) was found between the composite resin materials tested in percentage weight loss. The resin composites Filtek P 90 presented an increase in percentage weight loss after final tooth-brushing cycles compared to the rest of the materials. The results of the roughness analysis (Table III) indicated that material type significantly influenced ($p < 0.01$) the resulting mean roughness of the composite resin materials tested. Brushing significantly increased roughness (Ra) ($p < 0.05$) for all composites. Filtek Z250, after brushing, was significantly rougher than the other resins followed by Filtek P 90, Spectrum TPH 3 and Filtek Z350 XT. SEM images (Figures 1,2,3,4) revealed the wear pattern of each material: tooth brushing caused severe change of surface topography of 'sub-micron hybrid' specimen compared to each other. Other composites behaved similarly. Clinically loss of gloss and also wear of surfaces could be consequences of the finding.

Discussion

The present study evaluated the short-term effects of wear and surface roughness of four different composites at initial and three tooth brush abrasion cycle intervals. Wear is defined as loss of material substance resulting in loss of anatomic form [10]. Besides the wear produced by abrasion, there are other types of wear generated by corrosion and fatigue [11]. Wear of composite resin restorations may result from degradation of the resin matrix, loss of filler due to faulty joining with the organic matrix through shear of exposed and cracked particles and cracks in the matrix

Table III. Mean surface roughness (micrometers) of tested composites after different tooth brushing cycles.

Material	Initial roughness (Ra)	Roughness after 5000 cycles (Ra 5)	Roughness after 10 000 cycles (Ra 10)	Roughness after 20 000 cycles (Ra 20)	Difference in roughness
Filtek Z 250	0.154^{A}	0.183^{B}	0.217^{C}	0.261^{D}	-69.48^{b}
Spectrum TPH 3	0.196^{A}	0.210^{B}	0.232^{C}	0.266^{D}	-35.71^{a}
Filtek Z 350 XT	0.191^{A}	0.209^{B}	0.223^{C}	0.257^{D}	-34.55^{a}
Filtek P 90	0.187^{A}	0.2^{C}	0.226^{C}	0.256^{D}	-36.89^{a}

Same upper case letter within a row indicates no difference (paired *t*–test, $p < 0.05$).
Same lower case letter within a column indicates no difference (Tukey's test, $p < 0.05$).
$n = 6$.

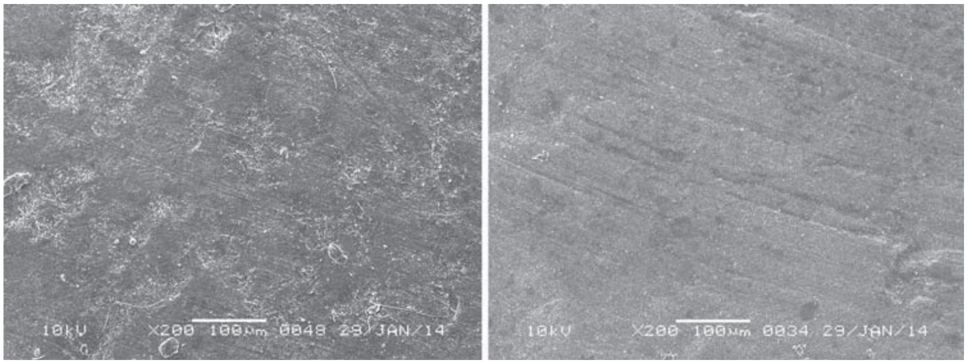

Figure 1. Scanning electron microscopy image (×200 original magnification) of Filtek Z250 Microhybrid specimen (before and after final toothbrush abrasion).

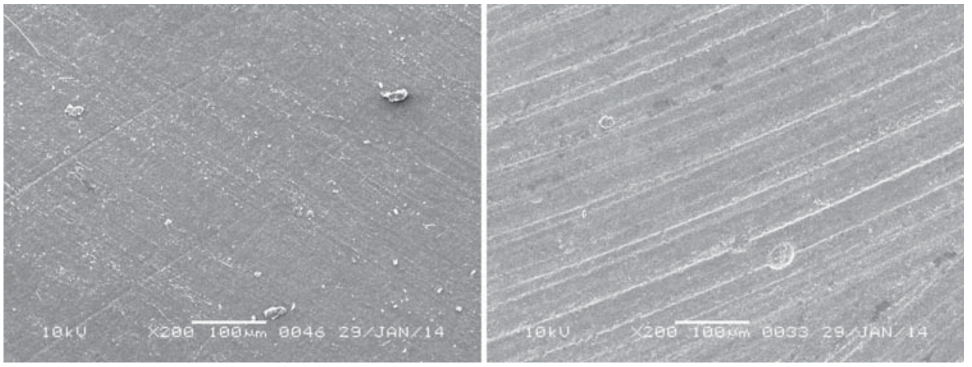

Figure 2. Scanning electron microscopy image (×200 original magnification) of submicron hybrid specimen (before and after final toothbrush abrasion).

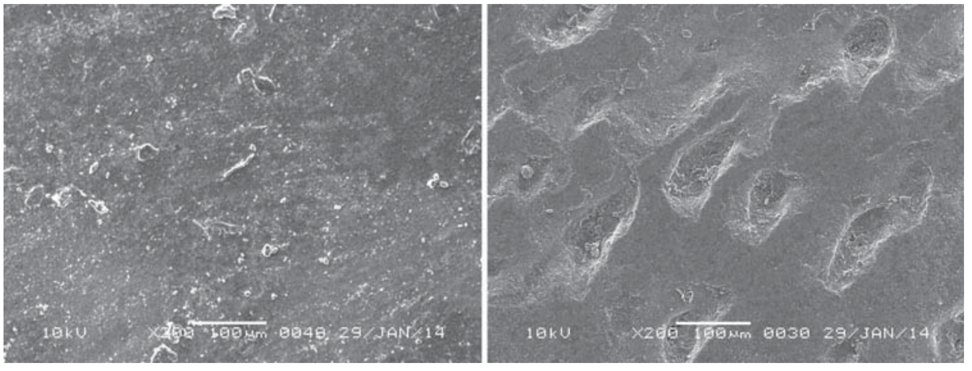

Figure 3. Scanning electron microscopy image (×200 original magnification) of Filtek Z350 Nano filler specimen (before and after final toothbrush abrasion).

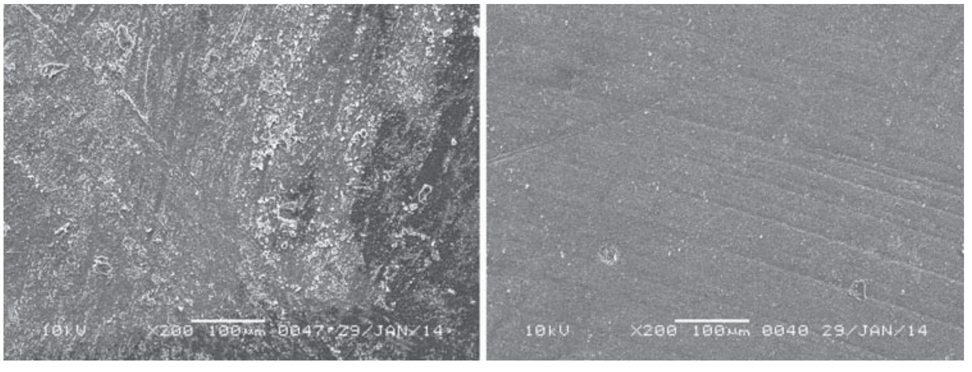

Figure 4. Scanning electron microscopy image (×200 original magnification) of Filtek P90 Microhybrid specimen (before and after final toothbrush abrasion).

[12-14]. Wear testing of dental materials utilizes commonly testing systems such as DIN, Acta, Zurich, Alabama, Freiburg, Minnesota, OHSU and Newcastle, which are typically two or three body wear tests [15]. These wear simulators cause stress more predominantly to the exposed and protruded filler particles rather than the polymer matrix. In this study we used a tooth brushing simulator to demonstrate wear behaviour, which was expected to induce more wear to the matrix and less to filler particles. A tooth brush/dentifrice abrasion concept works on programmable brushing techniques and motions, dentifrice abrasive slurry and number of cycles, time and load.

A clinical evaluation of these materials is exigent since some factors such as dietary habits and chewing forces might influence the results [11,16,17]. The majority of *in vitro* studies designed for assessing the surface integrity of restorative materials under conditions simulating the chemical environment of the oral cavity have involved mechanical forces, especially chewing simulation [18-22]. However, it is also pertinent to determine how restorative materials perform as a consequence of brushing abrasion, since this is the main cause of material loss that restorations encounter in non-stress locations [23]. It is well documented that the type of toothbrush used has no effect on abrasion when used with water, however some effect on wear when carrying toothpaste. Soft brushes cause reportedly more abrasion than medium or hard brushes, which is explained with increased retention of toothpaste by smaller diameter filaments and denser tufts on soft brushes and the greater flexion of filaments, increasing the area of contact with the surface [24]. This was in agreement with the results which show slightly higher roughness values compared to the normal. The cycle intervals of 5000, 10 000 and 20 000 in the study represented 6 months, 1 year and 2 year of tooth brushing wear with the assumption that 10 000 strokes simulate ~1 year of tooth brush wear [25]. The brushing force of 200 g applied in our study was well within values as recommended by ISO/TR [26]. During mastication, wear leads to dislodgment of filler particles which result in surface voids on the surface of restoration, exposing the organic matrix to the oral cavity. Thus, the resulting wear surfaces have more voids with the large filler [27]. The present study showed Spectrum TPH 3 had the lowest weight loss followed by Filtek Z 250, Filtek Z350 XT and Filtek P 90. This can be well substantiated with the fact that round shape of fillers in submicron hybrid composite is responsible for integrity and esthetics of this composite, which offers the clinician high resistance to abrasion [28]. Aker [11], in one of his studies, confirms that the size of filler particles can affect the resistance to abrasion. Furthermore, the author suggests that the volume loss is caused by the combination of the removal of the matrix and the eventual dislodgment of some particles as the matrix is worn away and the big particles seem to hinder that removal.

It is known that profilometer is the most commonly used method of evaluating [8]. Profilometer gives the quantitative measures of surface irregularities [29]. In the present study, the Filtek Z350 XT had lowest surface roughness values and the highest was found in Filtek Z 250. These values are in good agreement with the concept of Resin composites with different classifications having different wear mechanisms that are influenced mostly by the filler systems in the materials [30]. Nanocomposites have small primary filler particles, so that the filler and resin matrix are abraded off together during wear. For microhybrid composites whose average filler particle sizes are ~1 μm, the relatively soft resin matrix is worn first and the inorganic filler stands out above the surface [31]. Wear could also be related to loosening of hybrid composite particles of actual composite resin. Although the average cluster size of the nano-composites is similar to that in microhybrid composite fillers, nano-sized primary particles in the nano-composites wear by breaking off individual primary particles. Thus, the resulting wear surfaces have smaller defects and better gloss retention [31]. The same concept of filler particles substantiates the high surface roughness value of Filtek Z 250 and Filtek P90 in the present study. Roughness values greater than 0.2 μm might result in a simultaneous increase in plaque accumulation and increased risk of secondary caries and periodontal inflammation [32]. In all the materials tested in the present study, the mean Ra values were greater than 0.2 μm. In one of the studies, Mandikos et al. [33] observed a significant correlation between depth of wear and mean of surface roughness of some indirect composite resins after tooth brushing. They also suggested that the differences in wear, hardness and surface roughness could occur due to differences in the chemistry or in the light-curing methods.

Silorane-based composite (P90) contains irregular filler particles of quartz and yttrium fluoride of 0.01–3.5 mm in size and bound to the organic matrix by an epoxy-functional silane bonding agent through a silanization process, which is notably similar to the methacrylate restorative materials. The methacrylate-based composite (Filtek Z250 and Filtek Z350) had a similar organic matrix and differently sized inorganic particles bound to the matrix with methacrylate-functional silane. Filtek Z350 contains 20-nm nanoparticles of silica and nanoclusters of zirconia-silica that range from 0.6–1.4 μm in size and the Z250 contains zirconia-silica particles that range from 0.01–3.5 μm [34]. The hardness of composite resins is dependent on the filler type and content and it has been suggested to correlate with mechanical properties, such as abrasion resistance or polishability. Boaro et al. [35] measured degree of monomer conversion of silorane-based composite after polymerization and showed that conversion of silorane was lower than that of all other tested methacrylate-based composites, which was also demonstrated by Porto et al. [34]. However, degree of monomer conversion is not necessarily related to the wear resistance, some composite with a high degree of conversion may wear more than the same composite with a lower degree of conversion [36].

It is likely that water storage of test samples for a longer period of time could have increased the wear rate. By diffusion of water to the polymer matrix of the composite, water also penetrates to the silane coupling agent promoted interface of filler particle and polymer matrix. Silane forms a polysiloxane network between the inorganic filler and polymer matrix which is prone for hydrolysis. On the other hand, it is known that silane or other coupling agent promoted adhesion is difficult to obtain with zirconium dioxide. Therefore, more detailed investigation of the influence of the long-term effect of water and bonding interface of filler particle to the polymer matrix will be carried out in the future.

Conclusion

Within the limits of the present study, wear and surface roughness increased with each cycle interval for all the materials, one composite resin demonstrated a higher increase in surface roughness than the other three tested brands of composite resins. Not much difference was observed in the weight loss between tested samples.

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