

IN VITRO WEAR OF THREE BULK FILL COMPOSITES AND ENAMELJean-François Roulet^{1*}, Nader Abdulhameed², Chiayi Shen³

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Cite this article:Roulet J-F, Abdulhameed N, Shen C. In vitro Wear of Three Bulk Fill Composites and Enamel. *Stoma Edu J.* 2017;4(4):248-253**ABSTRACT**

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Introduction: This in vitro study aimed at testing the hypotheses that (1) there is no difference in wear in vitro among 3 bulk-fill composites investigated and their respective antagonists, and (2) the tested bulk-fill wear is not different from enamel.**Methodology:** X-tra fil (Voco; [X]), Tetric-N-Ceram Bulkfill (Ivoclar Vivadent; [T]), QuiXX (Dentsply; [Q]), and enamel [E] specimens ($\varnothing=8$ mm, depth=1.5 mm, n=8/material) were subjected to wear in a chewing simulator (CS 4.8, SD Mechatronik) with steatite antagonists ($\varnothing=6$ mm). 1.2×10^5 cycles (0-49 N, 0.7 mm lateral movement, 1 Hz) were performed while simultaneously thermocycling (5/55°C) every 90 s. The volumetric wear of the materials was measured with a 3D laser scanner.**Results:** The total wear of bulk-fills was: [X]: 0.64 ± 0.07 mm³; [T]: 0.66 ± 0.08 mm³; [Q]: 1.58 ± 0.14 mm³. The total wear of enamel (0.24 ± 0.03 mm³) was significantly lower than that of the bulk-fills ($p < 0.0001$). The total wear of the antagonists was: [X]: 0.32 ± 0.02 mm³; [T]: 0.24 ± 0.04 mm³; [Q]: 0.27 ± 0.02 mm³; [E]: 0.12 ± 0.01 mm³. The wear of the antagonists by [X] was significantly higher than by [T] and [Q] ($p < 0.001$). Enamel produced the lowest wear of the antagonists ($p < 0.0001$). The wear was linear between 5×10^3 and 1.2×10^5 wear-cycles. A negative correlation between the wear of the composite materials and that of the antagonists was found.**Conclusion:** In vitro wear of Tetric-N-Ceram Bulkfill was in the expected range and equal to X-tra fil. QuiXX wear was 2.7 times higher. The antagonist wear was significantly lower, less than 50% of the wear of the composites and the enamel. Both hypotheses were rejected.**Keywords:** chewing simulator, wear, bulk-fill composite, thermocycling, enamel.**1. Introduction**

Approximately 5 years ago a new category of resin composites was introduced for bulk-filling deep and wide dental cavities. These new materials, called bulk-fill composites, are claimed to provide a faster and easier procedure than the traditional incremental restoration technique.¹⁻⁷ This innovation was introduced following the general marketing trend for faster, easier and more convenience in restorative dentistry. Bulk-fill resin composites are claimed to be placed up to 4 or 5 mm thick increments (bulks) skipping the time-consuming layering process, and cured with light exposure time of up to 20 s.⁸ To accomplish this, the well-known and clinically proven resin chemistry and filler technology had to be modified in several aspects. The translucency of the material had to be increased to allow the blue light-curing wavelength to penetrate to the required depth of the material.⁹ It was accomplished by either using less pigments and/or by matching the refractive index of the resin as closely as possible to those of

the fillers in order to minimize the light scattering at the resin-filler interface.⁸ Another possibility was to use more effective photo initiator systems (e.g. germanium-based light-initiators, such as Ivocerin, Ivoclar Vivadent AG, Schaan, Liechtenstein), which allow the composite to be cured with less light energy per cm².¹⁰

Furthermore, for bulk-fill composites it is beneficial to reduce the polymerization shrinkage stress, to reduce the stress challenge to the tooth-restoration interface, thus allowing a good seal of the restoration by the adhesive system. One way to accomplish this is to minimize the resin content of the composite by using rather coarse fillers (particle sizes much higher than 5-10 μ m). Since the surface area of such particles is smaller, less resin is needed to wet it.¹¹ However, if this is done with conventional glass fillers, the surface characteristics and thus the polishability deteriorate.¹² A way around this is to use composite fillers having almost the same composition as the cured composite. They polish

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Professor Jean-François Roulet, Dr. med. dent, Dr. hc., Professor, Director of Center for Dental Biomaterials, Department of Restorative Dental Sciences, Center for Dental Biomaterials, College of Dentistry, University of Florida, 1395 Center Drive, Gainesville, FL 32608 USA; Phone +1 352 273 5850; Fax: +1 352 846 1643, e-mail: jroulet@dental.ufl.edu

as well as a micro hybrid composite.¹¹ A further optimization can be reached by using multimodal filler compositions.¹³ Another possibility is to use resins which shrink less (e.g. larger molecules, which means less double bonds to be reacted); however, these monomers should have a low viscosity in order to be able to wet the filler particles.¹¹ Finally, on the monomer side, longer spacers can be built between the reactive sites, which allow for stress relief after polymerization.⁸

Czasch & Ilie⁵ showed that curing Sure Fill SDR (DENTSPLY DeTrey GmbH, Konstanz, Germany) and Venus Bulk-fill (Heraeus Kulzer GmbH, Hanau, Germany) in 4-mm bulks for 20 s can be recommended based on FTIR and micro hardness data. Ilie et al.¹⁴ reported the same results with Tetric EvoCeram Bulk-fill (Ivoclar Vivadent) and X-tra base (VOCO GmbH, Cuxhaven, Germany). There is a growing body of literature demonstrating that the bulk-fill concept is validated, when considering curing depth, mechanical properties in the cervical area, and margin quality.¹⁻⁸ However, it is still not known if the modifications in the composition have an influence on the wear behavior of the bulk-fill composites. Therefore, the objective of the present study was to compare the wear behavior of different bulk-fill restorative materials as well as enamel *in vitro*.

The null hypotheses tested were: (1) bulk-fill composites show the same amount of wear and (2) the wear of composites is equal to the wear of the enamel.

2. Materials and Methods

The following bulk-fill materials were used: (X-tra fil, [X], Voco), (Tetric N-Ceram Bulk Fill, [T], Ivoclar Vivadent) and (QuiXX, [Q], Dentsply). Eight samples were prepared for each brand according to the manufacturer recommendations.

Thirty-two aluminum sample holders (inner Ø 7.9 mm, depth 1.5 mm) were grit-blasted with 27 µm aluminum oxide particles (EtchMaster Tips Small, Groman, USA), then one coat of universal primer (Monobond Plus, Ivoclar Vivadent) was added and left for 60 s, followed by air blasting to evaporate the solvent. Then one coat of adhesive (Optibond FL 2, Kerr, USA) was applied and light-cured for 10 s using the Bluephase G2 unit at "High" mode delivering 1450 mW/cm² and having a radiant exposure of 14.5 J/cm² at a distance of 1.5 mm (verified with MARC Resin calibrator, Bluelight Analytics Inc., Halifax, NS). The composites [Q], [T] and [X] were filled into 24 sample holders (n=8/each material) in one increment, then the top surface was flattened with a Mylar® matrix band and light-cured at high mode for 10s (Bluephase G2).

The composite surfaces were finished and polished by using silicon carbide discs (Sof-Lex, 3M, St. Paul, MN, USA), light orange disc for finishing and yellow disc for polishing, each for 15 s. All samples were then stored in distilled water at 37° C for 3 weeks.

Human enamel samples were obtained from extracted incisors, stored in 0.4% chloramine solution. The IRB1 of the University of Florida allowed the use

of extracted teeth, if they are completely anonymized (IRB.UF 201500060). They were mounted with adhesive technology as described above for the steatite antagonists on eight grit blasted aluminum sample holders for the chewing simulator CS 4.8 (Mechatronik, Germany), perpendicular to the long axis of the sample holder. They were then ground flat and polished using the previously described protocol and materials.

Steatite balls (Ø 6 mm) mounted into aluminum holders using resin composite were used as antagonists. One antagonist per sample (n=24) was used, and then discarded after finishing all cycles. The antagonists were scanned with (Laser scanner LAS-20, Mechatronik, Germany) before starting the experiment. The samples were randomly distributed to the chewing simulator chambers (CS-4.8,) using random numbers.¹⁵

The chewing simulator was run according to the parameters listed in Table 1.

The composite samples were scanned after each round (Table 2). However, the antagonists were scanned only prior to the experiment and at the end of the experiment.

This resulted in 1.2x10⁵ mechanical cycles and 1333 thermal cycles as a total.

Dedicated software (Geomagic® Control™ 2014, 3D Systems, Inc., Rock Hill, SC, USA), was used to analyze the scanned data. After each round, volumetric wear of the samples (composite and enamel) was determined by using the flat surface of the sample as a reference plane. With the "fill" command the software calculated the volume of the observed wear facet. The wear of the steatite antagonists was measured by superimposing the worn antagonist with the initial, unworn antagonist. Volumes under the reference plane were calculated using a common reference plane. The difference between the new and worn antagonist was considered to be the volumetric wear of the antagonist. Data were analyzed using ANOVA, linear regression and Tukey test after the normality of the data was confirmed with Komolgorov-Smirnov test (JMP, SAS, Cary NC, USA).

Table 1. Settings of Chewing Simulator.

Load	49 N
Upstroke	2 mm
Down stroke	1 mm
Horizontal movement	0.7 mm
Upward speed	60 mm/s
Downward speed	60 mm/s
Horizontal speed	40 mm/s
Frequency	1HZ
Thermocycling	5 °C-55 °C 30 s holding time, Transfer time 15 s, Total cycle 90 s
Direction	One way under load, back without load

Table 2. Number of mechanical cycles and time intervals for scanning.

Round No.	No. of cycles	Total
1	1x10 ²	1x10 ²
2	4x10 ²	5x10 ²
3	5x10 ²	1x10 ³
4	1x10 ³	2x10 ³
5	3x10 ³	5x10 ³
6	5x10 ³	1x10 ⁴
7	1x10 ⁴	2x10 ⁴
8	1x10 ⁴	3x10 ⁴
9	1x10 ⁴	4x10 ⁴
10	1x10 ⁴	5x10 ⁴
11	1x10 ⁴	6x10 ⁴
12	1x10 ⁴	7x10 ⁴
13	1x10 ⁴	8x10 ⁴
14	1x10 ⁴	9x10 ⁴
15	1x10 ⁴	1x10 ⁵
16	1x10 ⁴	1.1x10 ⁵
17	1x10 ⁴	1.2x10 ⁵

Table 3. Wear rate of the tested materials. Same superscript letters mean same statistical group ($p < 0.001$).

Material	Wear rate (x10 ³ μm ³ /cycle)	
	Mean ± SD	Stat. Group
Enamel	2.23±0.28	A
X-tra	4.64±0.41	B
Tetric	5.38±0.63	B
QuiXX	11.19±1.12	C

3. Results

One-way ANOVA of the volumetric wear of the composites and enamel, and respective wear of the antagonists after 1.2x10⁵ load cycles showed there were statistically significant differences between the four materials tested and the respective antagonists ($p < 0.0001$). The results of Tukey's wear ranking for each material and respective antagonist along with the mean and standard deviation of the wear are shown in Fig 1. With exception of QuiXX, the antagonist wear was less than half of the wear of the materials. The wear-loading cycle plots of each test showed that a linear correlation could be identified between volumetric wear and number of load cycles in the range of 5x10³ to 1.2x10⁵ load cycles. Linear regression was performed for each test and the slope of the line was considered as the wear rate of each individual test. The degree of fit of linear regression

was greater than 98% for each specimen. ANOVA of the wear rates showed there was statistically significant difference between the tested materials ($p < 0.0001$). Fig. 2 shows the plot of mean wear vs the number of loading cycles. The straight lines associated with each material are the result of linear regression based on the mean wear values of 8 specimens for each material. The mean wear rates along with the standard deviation and Tukey's ranking are shown in Table 3. The wear behavior of Tetric N Ceram Bulk Fil and X-tra fil were almost identical, while QuiXX showed statistically significant more wear.

4. Discussion

4.1. Methodology

The new bulk fill materials selected for this study were from the group of the high viscosity ones, designed to be used as a regular filling material, but being easier and faster in their application, and thus being exposed to the occlusal stress of antagonists.

When looking at mechanical properties of composites, it is important to make sure that the composite is well cured, which means that the polymer network has reached its optimal degree of conversion. Thus one can expect the best possible mechanical properties of the material. Therefore, in this study it was decided to use the curing times recommended by the manufacturers. Furthermore, the total energy delivered to the composites was determined to be 14.5 J/cm². It is in agreement with the recommendations found in literature, showing that to adequately cure resin composites of 2-mm thickness, between 12 and 24 J/cm² of energy is needed.^{16,17}

Wear is a very complex process; therefore, there is no single standard procedure for wear testing. Many wear testers use different approaches; however, lately two-body wear with a sliding component and preferably computer controlled forces and movements are the preferred approach.¹⁸

Since every wear tester has a different operational approach¹⁸ different antagonists regarding material, shape and dimensions are used.¹⁹⁻²⁴ In the present study steatite antagonists with a spherical shape and 6 mm diameter were used due to their hardness, reproducibility, standard form similar to a cusp, and easy availability. Furthermore, most chewing simulator users prefer these antagonists which allow better comparisons with other studies. Standard parameters were used for operating the chewing simulator. Therefore, our data are well comparable for instance with the ones obtained by the Ivoclar Vivadent group in Schaan.¹³ The slight difference in the measured wear between the Ivoclar group and the values presented in this study may be explained with the different antagonists used. In the present experiment spherical steatite antagonists were used, while Ivoclar Vivadent used standardized Empress (leucite ceramic) antagonists which had the shape of a molar cusp.¹³

The wear values obtained in this experiment were almost half as high as the ones obtained with similar composites in a former experiment using the same chewing simulator.²⁵ This difference may be explained by the different loads used. In the present experiment 49.05 N load was used as others do,

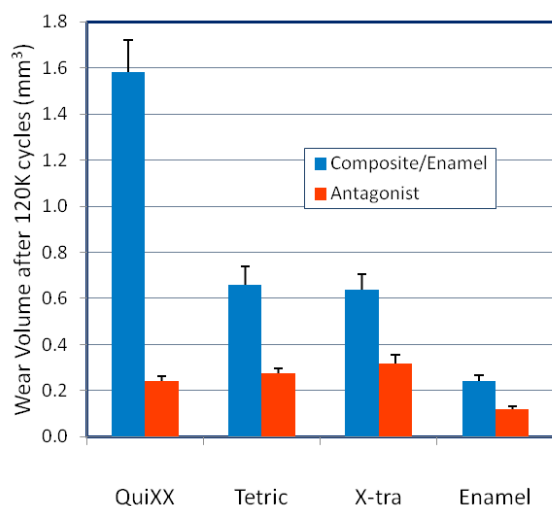


Figure 1. Total wear of 3 Bulk-fill composites and enamel (control) as well as the steatite antagonists after 12×10^5 cycles. Letters below the columns indicate Tuckey's group.

while in the former experiment the load was 58.86 N, which seems to be too much since fractures of the samples had occurred. It is difficult to determine the actual chewing force under function. Literature data show high variability (20 - 120 N). The decision to use 49.05 N was based on a paper by Gibbs et al.²⁶ who reported that value to be the average chewing force under normal function.

To measure the wear facets a laser scanner was used. Heintze et al.²⁷ have indeed shown that there was no significant difference between a mechanical or optical profilometer and a laser scanner.

As in a former experiment,²⁵ the wear behavior in the first 5×10^3 cycles was inconsistent and had a higher variability. This is a known effect called "running in". Therefore, the analysis of the data began at 5×10^3 cycles. From that point on, the wear development was linear with an excellent fit ($R^2 > 0.98$; Fig 2), which confirms the findings from Heintze et al.,^{21,27} Wang et al.²⁸ and Matias et al.²⁵ It allows to calculate a wear rate (= volume loss/cycle) which is best expressed in $\mu\text{m}^3/\text{cycle}$ (Table 3). It is thus possible to make direct comparisons independently from the number of cycles run. Nevertheless, it is recommended to run at least 1.2×10^5 cycles, to exclude an unnoticed change in the slope of the wear rate. The latter may be in fact due to fatigue-induced catastrophic failure, as we have seen with a glass ionomer cement (unpublished data).

4.2. Results

With the exception of QuiXX, the results of this study can confirm Heintze's,^{21,27} and Matias's et al data²⁵ that the antagonist's wear is about half the wear of the composite materials (Fig. 1). This may be explained by a different composition of QuiXX compared to the other composites, especially in its glass filler. That filler seems to be less hard than the fillers used in the other composites, thus being easier abraded, but at the same time being gentler with the antagonists.

QuiXX was worn three times more than the other two materials ($p < 0.001$). Looking at the composition of QuiXX as indicated in the directions for use, one can see that besides UDMA and TEGDMA, Di-

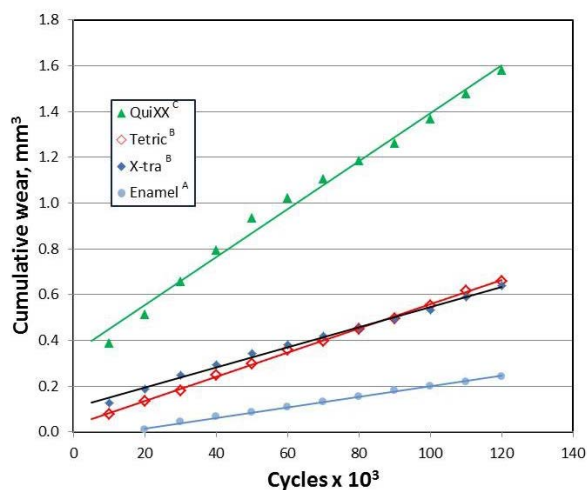


Figure 2. Linear regression of wear vs cycles for the tested composites and enamel ($p < 0.0001$). Superscript letters next to the name of the materials in the legend show Tuckey's group.

and Trimethacrylate resins, also a Carboxylic acid-modified dimethacrylate resin has been added. In addition, silanated strontium aluminum sodium fluoride phosphate silicate glass was used as filler. Furthermore, the material is delivered in a blister, obviously to prevent a ionic reaction between the carboxylic acid hydrolyzed by water that may diffuse into the material and the glass, which would make the material harden in its package. The manufacturer claimed fluoride release as well. Both facts lead to the suspicion that compomer technology was used for that product, and that could at least partly explain the increased wear of that material.²⁹⁻³²

X-tra fill is characterized by the manufacturer as a hybrid composite with 70.1% vol filler content and Bis-GMA, UDMA, BHT and TDMA as resins. Multimodal filler distributions with prepolymerized composite particles have been used for that material. Similar composition can be found in Tetric N Ceram Bulkfil, which could explain the same wear behavior. It is not known by the authors, if Voco uses similar filler technology. Looking at the antagonist wear one may speculate that the filler used by Voco might be of a conventional type, and in average coarser than the one used in the bulk-filled material by Ivoclar Vivadent. An interesting fact, the composite with the highest wear (QuiXX) has worn the antagonists the least and the composite with the least wear (Xtra) has worn the antagonist the most. This could be partly explained by the particle size, particle size distribution, the properties of the fillers (composition, hardness) and the filler load. If the particle distributions and the composition of the fillers used were known, this statement could be verified.

Besides showing the least wear, enamel also showed the least antagonist wear. This can be explained with the structure of enamel, which is very dense. The size of the hydroxyapatite crystals is much smaller than the ones of the fillers used in the tested bulk-fill composites. Once polished, the enamel surface is very smooth and generates low friction.

Since there are considerable differences in the

different wear testing devices, it is not possible to directly compare the volumetric wear data from different approaches. Therefore, only studies done with Willitec/Mechatronik wear testing machines can be used to do direct comparisons with the present study. Lazaridou et al.³³ found for Tetric EvoCeram 0.33 mm³, while Tetric N Ceram Bulk-fill showed 0.66 mm³ in the present study, which is substantially higher. Differences in the methods may explain these different findings. Lazaridou et al were loading the samples in water at 37° C, while in the present study the samples were thermocycled, which represents an additional stress.

Heintze et al 2006²⁷ have used almost the same approach as used in this study and measured for Tetric N Ceram Bulk-fill, approx. 0.6 mm³. D'Arcangelo et al.²² reported mean wear values for different direct composites between 0.529 ±0.139 mm³ and 1.425±0.245 mm³. However, they used a different antagonist material (zirconia) and shape (round tip 3 mm diameter). Hahnel et al.³⁴ measured the wear of 16 different resin-based restorative materials and found that the wear of Quixfil was approximately three times that of Tetric Ceram, which confirms the findings of this study.

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As all materials that have crosslinking in the resin matrix, flowable composites express some viscoelastic properties.³⁵ Thus, bulk-fill composites are not exempt from this property, as has been shown by Papadogiannis et al.³⁶ Stressing the composite in the chewing simulator may have created some creep, which could be seen as a confounder of the true wear that was measured in the present study.

5. Conclusions

In vitro wear of Tetric N Ceram Bulkfil was in the expected range and equal to X-tra fil. The wear of QuiXX was 2.7 times higher. Enamel was worn the least. The antagonist wear was significantly lower, less than 50% of the wear of the composites and the enamel.

Author contributions

Equal contribution to the paper.

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The authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

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Questions

Bulkfill composites

- a. can be cured up to 8 mm thickness in 5 seconds
- b. can be cured in 4-5 mm increments in up to 20 seconds
- c. can be used in one increment for every restoration
- d. have a very low translucency

In the experiment, the load of the chewing simulator was

- a. 29 N
- b. 39 N
- c. 49 N
- d. 59 N

The wear of enamel after 120'000 cycles was

- a. significantly less than the one of the composites
- b. significantly more than the one of the composites
- c. equal to the one of the composites
- d. 8x higher than the wear of the composites

The wear of the antagonists after 120'000 cycles was

- a. equal for all tested composites
- b. significantly lower than the wear of the composites
- c. significantly higher than the wear of the tested composites
- d. the same as the one of the tested composites