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In vitro wear of two bioactive composites and a glass ionomer cement

Objective of the study:
to measure the in vitro wear of two bioactive smart composite restorative materials and one glass ionomer cement.

Materials and methods:
The smart composites Activa (Pulpdent) and Cention N (Ivoclar Vivadent) and the glass ionomer cement Fuji IX (GC) were applied into aluminum sample holders, pressed against a glass plate and stored in water for 3 weeks after curing. The samples were subjected to 400,000 load cycles of 49 N in the CS-4 chewing simulator (Mechatronik) against steatite antagonists and subjected to 4,440 thermocycles from 5 °C to 55 °C. Samples were evaluated with replicas after 5,000, 10,000, 20,000, 40,000, 60,000, 80,000, 100,000, 120,000, 160,000, 200,000, 240,000, 280,000, 320,000, 360,000 and 400,000 cycles with a laser scanner (LAS-20, Mechatronik) and the Geomagic software (wear volume). The data was analyzed with ANOVA and Tukey test. Selected wear facets were analyzed with a scanning electron microscope (SEM).

Results:
The increase in wear was almost linear and after 60,000 cycles significantly different depending on the material (Activa < Cention N < Fuji IX). After 400,000 load cycles the following wear was measured: Activa 1.571 mm^3, Cention N 2.455 mm^3 and Fuji IX 5.622 mm^3. The wear of the antagonist was slight and in the reverse order ($p < 0.001$): Fuji IX 0.021 mm^3, Activa 0.091 mm^3 and Cention N 0.126 mm^3. SEM analysis showed pores in the powder-liquid systems. The composite and their antagonists had scratched surfaces, something that was not seen on the glass ionomer cement.

Discussion:
The bioactive composites that were tested had wear values comparable to the modern hybrid composites determined by the authors with the identical test method. The lesser wear of Activa in comparison to Cention N can be explained by the fact that the latter material is designed as a powder-liquid system with manual mixing.

Conclusion:
Based on their wear behavior the tested bioactive smart composites are suitable for posterior fillings (as an amalgam replacement) while the great wear to the glass ionomer cement confirms this indication (non load-bearing class I and II fillings).

Keywords:
smart composites; alcasites; glass ionomer cement; in-vitro-wear

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Translation: Jacobi Übersetzungen
Citation: Roulet JF, Hussein H, Abdulhameed NF, Shen C: In vitro wear of two bioactive composites and a glass ionomer cement. Dtsch Zahnärztl Z Int 2019; 1: 24-30
Peer-reviewed article: submitted: 13.11.2017, revised version accepted: 14.05.2018
DOI.org/10.3238/dzz-int.2019.0024-0030

Name	Type	Manufacturer	Charge #
Activa	Smart composite	Pulpdent, Watertown MA 02472 USA	160615
Cention N	Smart composite	Ivoclar Vivadent Schaan FL-9494	U19921
Fuji IX GP	Radiopaker glass ionomer cement	GC Tokyo, Japan	1604121

Table 1 Materials used

Introduction

Composites have been improved continuously since their invention in the 1950s [1–3]. But this has happened without abandoning their fundamental concepts [12]. Most of the improvements took place in the filler technology. In parallel to the improvement of the milling technology is was realized that an optimal 'smart' distribution of the filler particle sizes caused a reduction of the share of the resin content, which had a positive effect on the polymerization shrinkage behavior [27].

Diacrylates continue to be used on the resin side, whereby many different monomers with widely differing molecular weights are used [27].

A new generation has been developed recently that is termed bioactive or 'smart' [20]. This name aims to communicate that these materials are capable of reacting to environmental conditions. If the pH drops then these materials release ions that can both neutralize the acids produced by the bacteria in the biofilm and are available for remineralization processes as well [29]. This is achieved through the use of acid-soluble glass in combination with new types of monomers that can be polymerized as diacrylates as before [30].

As only little data can be found in the literature on Activa (Pulpdent) [6, 24], we will provide a brief description of this new class of composite, named Alcasite [11, 30] based on the example of Cention N (Ivoclar Vivadent). The fillers in this composite comprises proven components (barium-aluminum glass, calcium-barium-aluminum-fluoride silicate glass, yterbium trifluoride and isofillers [prepolymerized particles]) [30]. A calcium-fluoride silicate glass is added as an active component that can release ions as a function of the acidity of the environment. This filler mixture powder, which also contains parts of the initiator system, is mixed with a diacrylate mixture consisting of urethane dimethacrylate, TMX urethane dimethacrylate [22], a short chain diluent monomer (tricyclodecane dimethanol dimethacrylate) and an hydrophilic dimethacrylate (polyethylene glycol dimethacrylate) for improved wetting of the tooth structure [30].

Load	49 N
Upward movement	2 mm
Downward movement	1 mm
Horizontal movement	0,7 mm
Speed of upward movement	60 mm/sec
Speed of downward movement	60 mm/sec
Speed of horizontal movement	40 mm/sec
Frequency	1 HZ
Alternating temperature bath	5–55 °C; 30 sec holding time, transfer time 15 sec, total cycle length 90 sec
Direction	Forwards under load, backwards without load

Table 2 Settings on the chewing simulator

The cured material is capable of releasing Ca^{2+}, F^- and OH^- ions as a function of the acidity of the environment because of their composition. The OH^- ions neutralize the acid while forming water, the calcium and fluoride ions can form calcium fluoride and together with phosphate ions calcium phosphate can be provided for the remineralization of enamel. This effect was documented in vitro up to 100 µm from the filling-enamel-interface [30].

With regard to the mechanical properties Cention behaves for the most part in the same way as a nano hybrid composite [30]. Its bending strength remains stable within the range between 100–120 MPa when stored in water (measured for up to 3 months). The same is true for the modulus of elasticity which is in the range of around 5 GPa [11]. Thus, Cention N fits into the range of known and clinically proven hybrid and nano hybrid composites [12]. The mechanical data for Cention N are also comparable with those of Bulkfil composites [11]. But Cention N can be applied more easily. The material is offered as a powder-liquid system for

Material	120,000 cycles	400,000 cycles
Activa	0.54875 ± 0.06151	1.57125 ± 0.22787
Cention N	0.95000 ± 0.15946	2.45500 ± 0.24202±
Fuji IX	3.05000 ± 0.31491	5.62250 ± 0.54706

Table 3 Wear in mm^3 after 120,000 and 400,000 load cycles (mean ± standard deviation); ($p < 0.0001$)

manual mixing. It is intentionally positioned by the manufacturer (Ivoclar Vivadent) as an amalgam replacement material on permanent teeth as well as a replacement for glass ionomer cement on deciduous teeth, particularly in countries where simple dentistry is required. The alternative to amalgam in these countries is glass ionomer cement.

The conceptual structure of Activa is similar to that of Cention N. It is provided as a 2-paste system in a static mixer, displays a bending strength of 105 MPa [6] and is comparable to Cention/N with regard to its mechanical properties. Both materials are auto-curing (amine peroxide 2-component system), but can also be photo-polymerized [24, 30]. As the material Cention N is relatively new, comparatively little is known about its wear behavior. Thus the objective of this study was to measure the wear of Cention N in vitro in comparison to a competitor product with similar composition and a classic glass ionomer cement (control).

Material and methods

The materials that were used are summarized in table 1. The production of samples took place at room temperature (approx. 21 °C) in accordance with the manufacturer's recommendations for each product. Activa (Pulpdent, Watertown MA 02427 USA) was applied with the Activa-Spenser and static 5 ml automix syringe (Pulpdent) in aluminum sample holders that were sandblasted and pretreated with Adhese Universal (Ivoclar Vivadent, FL 9494 Schaan Liechtenstein). Then a mylar matrix was laid on the material and the surface pressed flat with a glass plate. The material was cured for 10 minutes (autocuring, no light curing!). Then polishing was carried out with Soflex discs (3M Espe, St. Paul, MN 55144 USA).

Two measuring spoons of powder and 2 drops of resin of Cention N (Ivoclar Vivadent) were applied to a mixing pad and mixed manually to a smooth consistency. First the liquid was mixed with half of the powder until it was well wetted and then the remaining powder was added in small quantities. The mixing time did not exceed 60 seconds. Then the paste was placed in the sand blasted and pretreated aluminum sample holder with a spatula, covered with a mylar matrix and pressed to a flat surface. The material was left for 10 minutes from the start of mixing (no light curing!). Then it was polished using Soflex discs (3M Espe, St. Paul, MN 55144 USA).

Two measuring spoons of powder and 2 drops of liquidof Fuji IX (GC, Tokyo, Japan) were applied to a mixing padand mixed manually to a smooth consistency. First the liquid was mixed with half of the powder until it was well wetted and then the remaining powder added in small quantities. The mixing time did not exceed 30 seconds. Then the paste was applied as described above in the pretreated aluminum sample holder. After 10 minutes the mylar matrix was removed and the surface polished with Soflex discs (3M Espe). Finally a layer of GC Fuji Varnish (GC) was applied to the surface. All samples were stored for at least 3 weeks in water at 37 °C before being subjected to wear.

Steatite antagonists (ø 6 mm, SD Mechatronik, D-83620 Feldkirchen-Westerham, Germany) were mounted with a light-cured composite in a pretreated aluminum antagonist holder as described above. New antagonists were used for each sample. The pairs of samples and antagonists were distributed on the chewing simulator chambers (CS-4, Mechatronik) using random numbers [25].

The chewing simulator was programmed in accordance with the parameters listed in table 2. The samples were simultaneously subjected to 4440 thermocycles of 5–55 °C.

After 5000, 10,000, 20,000, 40,000, 60,000, 80,000, 100,000, 120,000, 160,000, 200,000, 240,000, 280,000, 320,000, 360,000 and 400,000 load cycles impressions of the samples were taken with a hydrophilic polyvinylsiloxane (Virtual Light Body Wash Material, Ivoclar Vivadent) and standard small trays (ø 18 mm). They were poured with stone (Microstone, Premium Dental Stone, Golden, Whip Mix, Louisville, KY 40209, USA) and scanned with a laser scanner (LAS-20, Mechatronik). Impressions were taken on the antagonists at the start and after 60,000, 120,000, 200,000, 280,000 and 400,000 load cycles. They were poured with stoneas described above and scanned with a laser scanner (LAS-20, Mechatronik).

The wear measurement (volume) was carried out using Geomagic software as described by Matias et al. [18]. The same principle was used in order to measure the wear on the antagonists. The wear data was determined by 2 evaluators independently of one another (HH and NA).

The data was analyzed with the SAS program using the ANOVA and Tukey test (SAS© 9.4, Cary NC 27513, USA).

Scanning electron microscope (SEM) images were produced of selected samples in order to perform a qualitative assessment of the wear facets and their surface structure. The samples and antagonists were coated in gold-platinum in a Technic Hummer 22020 Sputter (Technics Inc, Alexandria VA 22310) for this purpose.

Results

The results of the material wear are shown in Figure 1 and Table 3. It is

clear that the wear of the glass ionomer cement was much greater at 400,000 load cycles (5.622 ± 0.547 mm³) than the one of the resin based based materials (Cention N 2.455 ± 0.242 mm³; Activa 1.571 ± 0.228 mm³). These differences were statistically significant (see table 3). No significant difference was determined between Cention N and Activa up to 60,000 load cycles (see Figure 1).

The antagonist wear is shown in Figure 2. It should be noted that Fuji IX ha d causedthe least abrasion of the antagonists (0.065 ± 0.0185 mm³). Both composite materials abraded the antagonists significantly more severely (Activa 0.156 ± 0.0239 mm³; Cention N 0.192 ± 0.013 mm³). This difference was statistically significant ($p < 0.0001$).

SEM images of the wear facets and the corresponding antagonists of the different materials are shown in Figures 3–5. It should be noted that both powder-liquid formulations (Cention N and Fuji IX) ended with pores in the structure (Figures 4 and 5) that were not found with Activa (static mixer in the paste-paste system) (Figure 3). The minimal wear of the antagonists by Fuji IX is confirmed by the surface structure of the antagonists. Hardly any damage is visible. Fuji IX only displayed small scratches on its surface. On the other side the composite materials displayed clear scratches on their wear facets and created similar scratches in the antagonists.

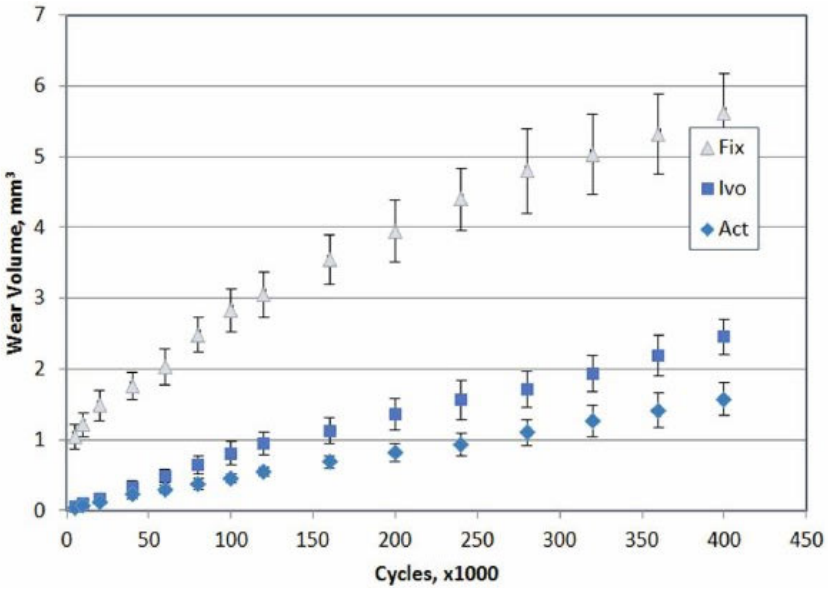

Figure 1 Wear of the investigated materials in mm³ (Fix = Fuji IX, Ivo = Cention N, Act = Activa); ($p < 0.0001$)

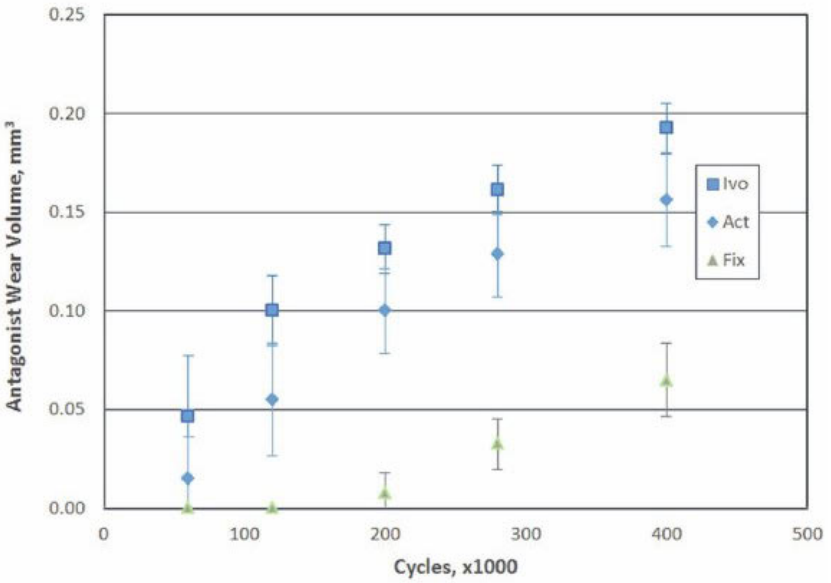

Figure 2 Wear of antagonists in mm³ against the materials tested (Fix = Fuji IX, Ivo = Cention N, Act = Activa); ($p < 0.0001$)

Discussion

If we look at the mechanical properties of composites then it is important to ensure that the composite is well cured and stable. The tested composite materials are auto-curing with light curing option [24, 30], in order to accelerate the application process. Light curing was purposely omitted in order to avoid a further variable that could distort the results, especially as Cention N was positioned for use in the markets with simple dentistry, where it can be assumed that light curing cannot be presupposed as standard.

Wear is a very complex process. That is why there is no specific standard for wear testing. It is particularly difficult to completely reproduce the clinical situation in vitro. The different in-vitro wear machines use different approaches; however machines with two-body wear and sliding component and preferably computer-controlled forces and movements have been preferred recently [13]. As every wear tester has a different approach to the work [13] different antagonists are used with regard to material, form and dimensions [4, 8, 14, 15, 18, 21]. In this study spherical steatite antagonists were used (ø 6 mm) because of their hardness, reproducibility, the standard form similar to a tooth cusp and easy availability. In addition to this most Mechatronik chewing simulator users use this antagonist, which allows comparisons with other studies. Standard parameters were used for the operation of the chewing similar. This makes our data easy to compare with those of the Ivoclar Vivadent Group in Schaan [16]. The

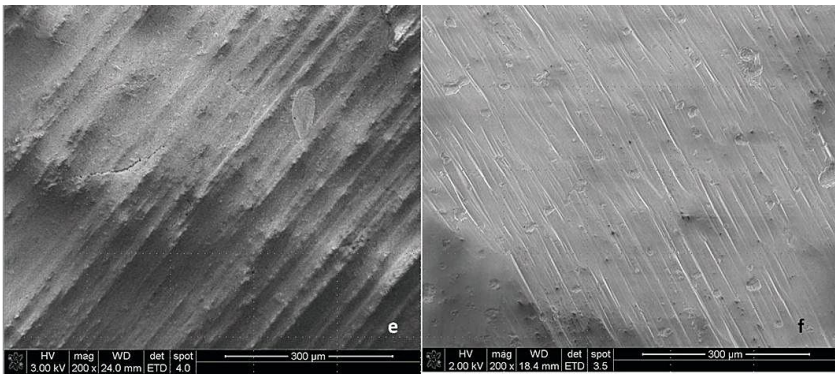

Figure 3 Activa wear facet after 400,000 load cycles (left) and corresponding antagonist (right). SEM 160x.

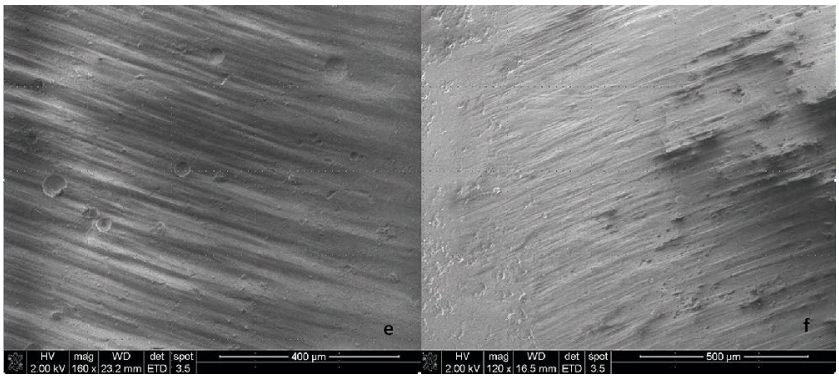

Figure 4 Cention N wear facet after 400,000 load cycles (left) and corresponding antagonist (right). Note the air bubbles in the material. REM, 160x (left); 120x (right)

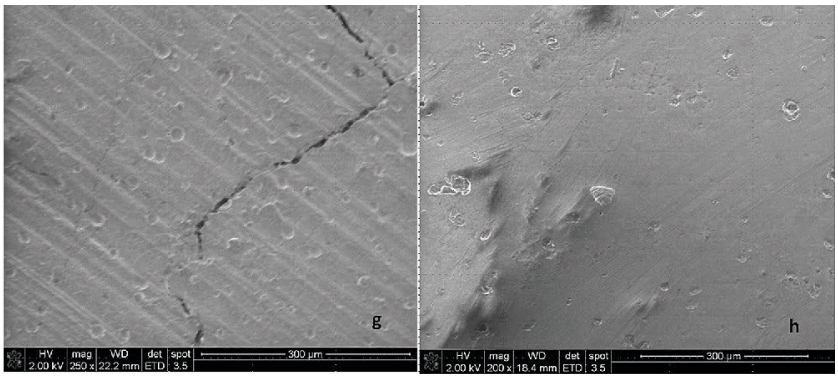

Figure 5 Fuji IX wear facet after 400,000 load cycles (left) and corresponding antagonist (right). Note the air bubbles in the material. Cracks are artifacts due to dehydration of the glass ionomer cement for sample preparation. SEM, 250x (left); 200x (right).

(Tables 1–3, Fig. 1–5: Roulet et al.)

slight difference can be explained by the different antagonists. Spherical steatite antagonists were used in this experiment while Ivoclar Vivadent used standardized Empress (leucite-ceramic) antagonists that are in the form of a molar cusp. The wear values that resulted from this experiment were only half of those achieved with similar composite materials in an earlier experiment [19] using the same chewing simulator. This difference can be explained by the different settings of the chewing simulator [19]. A load of 49 N was used in this experiment while the previous experiment used 59 N, which appears to be too much as fractures appeared in the samples. It is difficult to determine the actual chewing force in vivo. The data in the literature reveals great variations (20–120 N). The decision to use 49 N is based on a study by Gibbs et al. [7] that reported that 49 N is the average chewing force in normal functions.

A laser scanner was used to measure wear facets. Heintze et al. [9] showed that there is no significant difference between a mechanical or optical profilometer and a laser scanner.

This study was performed using almost the same study as earlier studies [10, 19, 28]. The difference was that the samples and antagonists that were used together in the Matias study were scanned directly while we decided to use hard plastic replicas. The reason for this was that we found distortions in the flat surface at the transition to the facet during the direct observation of facet in polished, flat composite or ceramic surfaces in the Geomagic software [5]. We also had 2 assessors who measured the wear based on the LAS 20 scans, which displayed identical data. This resulted in slight standard discrepancies overall, so that we were able to differentiate between the material wear in the different materials at an early stage (from 80,000 load cycles between Cention N and Activa).

However, the total number of cycles was increased to 400,000 in this study because the linear course of the wear changed at approx. 350,000 cycles in a pilot study with glass ionomer cement (unpublished data).

The smart composites Activa and Cention N both offer a light curing option. However, Cention N is directed towards emerging economies where it is generally unlikely that light-curing equipment will be available. Therefore, it was decided to only use these materials in auto-curing mode.

The wear behavior in the first 5,000 cycles was inconsistent and had greater variability, as in an earlier experiment [19]. This is a known effect known as ‘running in’. The analysis of the data therefore began at 5,000 cycles. From this point the wear development was linear with excellent correlation ($R2 > 0.98$; see Figure 2), which confirms the results of Heintze et al. [8, 9], Wang et al. [31] and Matias et al. [19].

When the wear volume was compared the smart composites with the bioactive properties had approximately the same values as Tetric N Ceram Bulkfil and X-tra fil, as tested in an earlier study [28]. At 120.000 load cycles Tetric N-Ceram had 0.66 ± 0.27 mm^3 and X-tra fil 0.64 ± 0.32 mm^3 wear. This compares well with this study, also at 120,000 load cycles, at 0.5487 ± 0.061 mm^3 (Activa) and 0.950 ± 0.159 mm^3 (Cention N). The data presented in this study is of the same order as the wear values (volume) submitted by Lendenmann and Wanner [16] for composites. The slight differences could be explained by the fact that different antagonists were used. Thus study used steatite spheres with a diameter of 6 mm while the Ivoclar-Vivadent method used Empress antagonists in the form of a natural cusp. The wear values of Cention N and Activa at 120,000 load cycles (table 3) correspond well to those of nanohybrid composites that had positioned themselves between 0.428 ± 0.083 mm^3 und 1.578 ± 0.37 mm^3 under identical conditions [10]. The mechanical data (bending strength and modulus of elasticity of the tested composite are over 100 MPa and around 5 GPa respectively [6, 11, 12], which corresponds to the values for the hybrid composites that are used routinely these days for posterior tooth fillings. The values are also substantially higher than required by the ISO norm (bending strength > 80 MPa).

Composites have shown excellent survival rates in use as posterior tooth fillings in long-term clinical studies. Lempel et al. [17] tested 4 composite materials in a well-controlled patient population in a retrospective study. After 10 years this showed a 0.1 % annual failure rate for Filtek Z250 and Herculite XR. For the products Gradia direkt and Renew this annual failure rate was 0.8 %. After 22 years of observation Da Rosa Rodolpho et al. [26] reported annual failure rates of 1.5 % for P-50 APC and 2.2 % for Herculite XR. Pallesen and van Dijken [23] tested P10, P30 and Miradapt as posterior fillings in a prospective study and found after 30 years an annual failure rate of 1.1 %. These experiments showed that composite materials with a bending strength of at least 100 MPa [12] function well in clinical application.

The very high wear rate of the glass ionomer Fuji IX confirms the limitation of the indication of this material for posterior tooth fillings in deciduous teeth and non load-bearing posterior fillings in permanent teeth. The low wear rate of the smart composites tested in this study makes them suitable for load-bearing posterior fillings given the bending strength of > 100 MPa. The simple method of application seems to make them very well suited to use as an alternative to amalgam fillings. The somewhat greater wear of Cention N can be explained by the smaller size of the glass filler, as can be seen in the scratch pattern on the SEM images (Figure 4). The differences in resin chemistry could also be responsible [24].

The clear difference between the glass ionomer and the smart composite materials can also be seen both in the surfaces and the materials themselves and in the corresponding antagonists (Figures 3–5). The composite materials and their antagonists display clear scratch marks that can be explained by the fact that filler particles (glass) are released under load and these could have served as an abrasive medium at times. The surface of the antagonist that wore the class ionomer cement and that was only slightly changed suggests that the glass used in the glass ionomer cement seems to be substantially softer than the glass in the composite materials.

The powder-liquid systems displayed air bubbles in the SEM images that were probably incorporated during mixing. It seems that the manual mixing of Cention N resulted in larger pores than the capsule mixing of the glass ionomer cement.

The SEM images of Fuji IX all displayed cracks (Figure 5) that must be classified as artefacts. It is known that glass ionomer cement displays severe cracks in the surface during drying.

Conclusion

The wear behavior of Cention N is in the same range for composites with the same chewing simulator. The wear rate was almost linear up to 400,000 load cycles. The wear of glass ionomer cement was 2.3 times greater than that of Cention N and 3.6 times greater than that of Activa.

From the point of view of wear behavior the positioning of Cention N as a filling material for posterior teeth is correct without limitations. Both tested composite materials have bending strengths above 100 MPa, which supports the above assessment. A capsulated material has somewhat better wear behavior because of its better and more homogeneous mixtures and smaller air bubbles and would therefore be desirable.

However, this data from one in-vitro experiment should be interpreted with caution and this should be validated with in-vivo studies!

Acknowledgements:
The authors thank Mrs. Margitta Hintz for proofreading the manuscript.

Conflicts of Interest:

Ivoclar Vivadent financed the study (contract ROULETARG50 dated 15.09.2016). The authors have no conflict of interest. The sponsor had no influence on the study design or the data collection and analysis. The decision to publish the study and the produce the manuscript were also taken independently of the sponsor.

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