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Original Article

An *in vitro* investigation of wear resistance and hardness of composite resins

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Abstract: *Purpose*: The aim of the present study was to investigate the wear resistance and hardness of five kinds of composite resins. *Materials and Methods*: Sixty-five specimens were fabricated with one nano-hybrid (Charisma Diamond), two micro-hybrid (3MZ250, Clearfil AP-X) and two packable (3MP60, Surefil) composite resins, according to a randomized complete block design (n=13, 8 for wear test; 5 for hardness test). The composites were filled in a rectangular mold, and light polymerization. After storage in 37 °C deionized water for 24h, all specimens were tested with a custom-made toothbrush machine with a stainless-steel ball as antagonist (3N loads, 1Hz, 6×10^5 cycles) immersed in calcium fluoride slurry. Wear volume, hardness and surface structure of each tested material was examined by a three-dimensional non-contact optical profilometer, Vickers indentation technique and scanning electron microscope. *Results*: The volume loss ranked from least to most as follows: Charisma Diamond, P60, Z250, Clearfil AP-X and Surefil. Regarding hardness, the rank from highest to lowest as follows: Clearfil AP-X, P60, Surefil, Z250, Charisma Diamond. The interactions between wear resistance and microhardness were not significant. *Conclusions*: The custom-made machine is considered suitable to simulate sliding of an antagonist cusp on an opposing occlusal composite restoration. Nanofilled composite may have superior wear compared to other composite resins.

Keywords: Composite resins, hardness, wear resistance

Introduction

Composite resins based on dimethacrylates and silane-coated inorganic fillers were introduced as dental restoratives in the mid-1960s [1]. Due to their excellent aesthetic properties, composite resins gain steadily importance and popularity for the restoration of all cavity classes. However, clinically, their relatively poor wear resistance is still considered as a factor that contributes to materials' failure [2].

Mair [3] defined wear as a consequence of the interaction between surfaces moving in contact, causing gradual remove of material. In the oral cavity, a lot of components contribute to the wear of enamel and dentin, such as the occlusal contacts to antagonist teeth (attrition), chewing on food items, toothbrushing with toothpaste or inhalation of dust (abrasion), acid attacks due to the consumption of acidic fruits and beverages, inhalation of industrial acids or vomiting and regurgitation of gastric juice, for instance, in bulimia and anorexia nervosa (erosion) cases [4].

Wear of composites is known to depend on filler particle-related features, particularly on the concentration and size of the filler reinforcement [5] and resin formulation [6] et al. Finer particles for a fixed-volume-fraction of filler have been documented to result in decreased interparticle spacing and thereby reduced wear [7, 8]. In terms of filler content, some in vitro wear studies have revealed that increased loading may enhance the wear resistance of dental composites [9-11]. As for the resin formulation, the study has shown that increasing resin viscosity generally lowers the wear resistance [12].

Ideal dental restorations should have wear resistance similar to that of tooth. Average clinical wear rate on occlusal contact areas of teeth

Table 1. Materials used in the test

Material	Type	Shade	Matrix	Filler type	Filler average size (μm)	Filler loading (vol%/ wt%)	Manufacture	Batch
Charisma Diamond	Nanohybrid	A3	TCD-DI-HEA, UDMA	Barium Aluminium Fluoride glass	0.005-20	64/81	Heraeus Kulzer GmbH, Germany	10035
Z250	Microhybrid	A3	Bis-GMA, UDMA, Bis-EMA, TEGDMA	Zirconia, silica	0.01-3.5	60/77.6	3M Espe, St. Paul, MN, USA	1370A3
P60	Packable	A3	Bis-GMA, UDMA, Bis-EMA, TEGDMA	Zirconia, silica	0.01-3.5	61/83	3M Espe, St. Paul, MN, USA	8100A3
Clearfil AP-X	Microhybrid	A3	Bis-GMA, TEGDMA	Barium glass, silica	0.1-15	70/86	Kuraray Medical Inc, Japan	01388A
Surefil	Packable	A	UDMA	Ba-Al-B-F-Si glass silica	0.8	58-66/82	Dentsply Caulk, USA	101220

Bis-GMA: Bis-phenol-A-diglycidylmethacrylate; BIS-EMA: bisphenol A-polyethylene glycol diether dimethacrylate; UDMA: urethane dimethacrylate; TEGDMA: triethylene glycol dimethacrylate; TCD-DI-HEA: 2-Propenoic acid, (octahydro-4, 7-methano-1H-indene-5-diyl) bis (methyleneiminocarbonyloxy-2, 1-ethanediyl) ester. *Composition as given by manufacturers.

is about 29μm per year for molars and about 15μm per year for premolars [6]. Another study showed at the mean pooled occlusal wear of four ultraviolet light-cured posterior composites at 17 years was 264μm, and that most wear (75%) occurred in the first 5 years [13]. Of course, results differ among evaluators due to operator variations, patient variations, evaluated products, and last but not least important, the wear evaluation method [14].

It is worthwhile to note that many factors besides wear can affect the lifespan of resin composites. However, it can be assumed that materials with better wear resistance should do better under cycling loading during normal occlusal and masticatory function. Therefore, the present study was to investigate the wear resistance and microhardness of five resin composites using a device for simulated toothbrushing, a three-dimension non contact optical profilometer and scanning electron microscope. The null hypothesis tested was that there would be no differences in wear resistance and microhardness.

Materials and methods

Materials

The five composite resins used are shown in **Table 1** together with their compositions. The applied matrix and filler concepts used in these materials are different.

Specimen preparation

Thirteen standardized samples (n=8 for wear volume loss measurement, n=5 for microhardness measurement) of each test composites were prepared. Unpolymerized material was applied to polytetrafluoroethylene molds (11mm length × 10mm width × 2mm depth), which were covered on both sides with polyester matrix strip and a rigid glass microscope slide. Composite resins were light cured (Woodpecker LED.F, 8mm diameter light-guide tip, Guilin Woodpecker Medical Instrument Co., Ltd, China; 1000mW/cm^2) from the top for 40s on each left and right halves and then turned over and identically cured from the bottom. The intensity of the curing light was verified before the polymerization using a curing light meter (CM-2500, Motion Medical Supplies & Equipment Corp, Taiwan). Rectangular stainless steel molds (17mm length × 12mm width × 5mm depth) were used for embedding specimens in acrylic resin. Specimens were retrieved from the mold and stored in deionized water at 37°C for 24h. Then each specimen was lapped with wet SiC paper, using consecutive grit numbers 600, 1500 and 4000. The samples were ultrasonically cleaned for 5min.

Wear testing

The wear testing was performed using a programmable logic controlled equipment (Xi´an Dongfeng Instrument Factory, China) [15] in

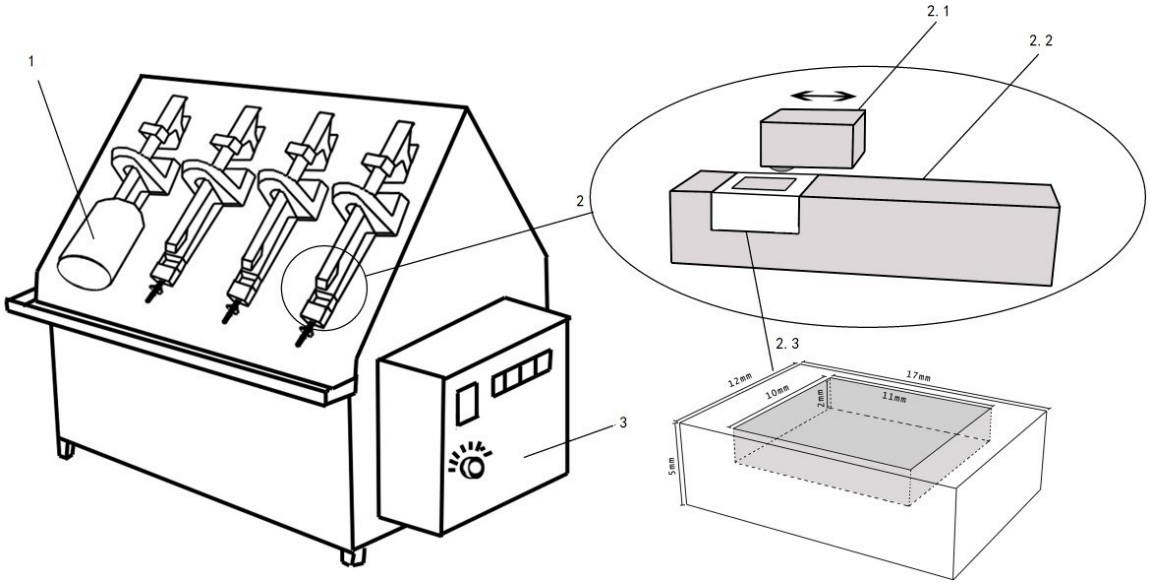

Figure 1. Schematic diagram of wear testing (1. acrylic chamber, 2.1 antagonist, 2.2 sample holder, 2.3 specimen, 3. control box).

Table 2. Surface profile measurement conditions

Device	Three-dimensional non contact optical profilometer (NANOVEA PS50 U.S.A)
Sampling interval	X:18μm; Y:50μm
Measuring range	8*7mm
Resolution of Z-axis	0.1μm

Figure 1, which allows for the adjustment for the frequency, number of cycles and duration of contact between the specimen and counterpart. The system can produce standardized load application in eight identical specimen compartments. Each compartment has a recess (17mm length × 12mm width × 5mm depth) where a specimen was positioned. Then a spherical antagonist made from stainless-steel (Ø=7.8mm) under a 3N load was applied to the specimens and moved across the surface over a 20mm linear path, generating abrasive wear at a frequency of 1Hz for a total of 6×10^5 cycles. Tests were carried out in eight individual compartments in the presence of 15ML fresh calcium fluoride slurry in an acrylic chamber [16]. Slurry and antagonists were renewed prior to each wear test.

Wear volume loss measurement (W)

After the test, specimens were cleaned with running water followed by an ultrasonic bath for 10min. The surface of each specimen was recorded using a three-dimensional non contact optical profilometer. **Table 2** shows the surface profile measurement conditions. Volume loss after wear test was measured by computing the volume of the worn area, which was at a level lower than the unworn surface level.

Microhardness measurement (H)

For hardness measurement, a microhardness tester (HXD-1000TM, Shanghai Taiming Optical Instrument Co., Ltd, China) was used. Vickers hardness numbers were determined from indentations made under 50g load for 15s by the arithmetic mean of three indentations randomly performed for each specimen and testing condition.

Scanning electron microscope examination (SEM)

One random sample of each composite material after 6×10^5 abrasive cycles was selected for SEM examination (Type Quanta 200 FEG, FEI Company, Netherlands). The samples were sputter-coated with gold and photographs were taken of representative areas at 1000× magnifications at 20.0kV acceleration voltage.

Statistical analysis

Data were analyzed using statistical software (SPSS 17.0 for Windows). Means and standard

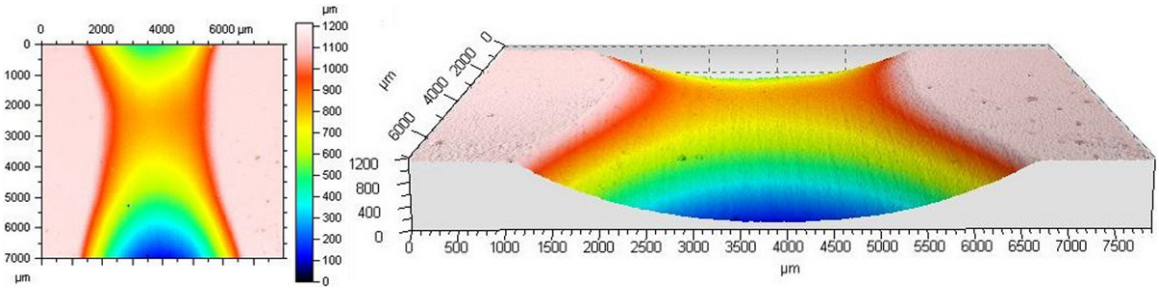

Figure 2. Two-dimensional and three-dimensional representation of the wear facet.

Table 3. Mean wear volume loss and microhardness of experimental composites (S.D.)

Resin composite	Wear volume loss (mm^3)	Microhardness (kg/mm^2)
Charisma Diamond	6.0057 (1.227)	54.89 (2.04)
Z250	8.4432 (2.4983)	79.27 (1.25)
P60	8.2588 (1.5561)	82.12 (4.12)
Clearfil AP-X	8.5956 (1.6379)	87.16 (2.16)
Surefil	10.8373 (1.9858)	79.37 (2.93)

deviations of W and H were calculated, and analyzed using one-way analysis of variance (ANOVA). Regression analysis was performed to investigate the relation between W and H. The level of significance was set to α=0.05.

Results

Wear facet observation

The wear facet of all the samples is consistent with the shape of the antagonist. Combined with a stereo microscope observation, the wear facet shows wear heavy in the middle and light on both sides, which is consistent with the result of the three-dimensional non contact optical profilometer (**Figure 2**).

W values and H values

Mean W values and H values are shown in **Table 3**. One-way ANOVA indicated significant differences in W between the materials (p<0.05). Charisma Diamond (6.0057mm^3) had the significantly lowest W, followed by P60 (8.2588mm^3), Z250 (8.4432mm^3) and Clearfil AP-X (8.5956mm^3) which did not differ from each other. Surefil (10.8373mm^3) had the significantly highest W.

One-way ANOVA indicated significant differences in H between the various materials (p<0.05). Significantly highest H was detected for Clearfil AP-X (87.16). P60 (82.12), Surefil (79.37) and Z250 (79.27) showed the intermediate values for mean H. Significantly lowest H was found for Charisma Diamond (54.89).

Regression analysis showed no significant correlations between W and H in **Figure 3** (P=0.0557).

SEM observation

Selected SEM of evaluated groups after wear testing was shown in **Figure 4**. The SEMs were taken from specimens in wear central. In general, the specimen surfaces of the five groups revealed observable differences from each other. Charisma Diamonds is characterized by a very smooth and uniformly worn surface. Small voids are seen throughout entire surface. The surfaces of Z250 and P60 show densely packed superficially abraded clusters in the surrounding resin matrix. The surfaces are quite uniformly abraded. The surface of Clearfil AP-X exhibit densely packed fillers with a wide grain size. Surefil presents more accentuated matrix degradation as well as more voids and cracks compared to others.

Discussion

Even though a laboratory study is not able to reproduce all the conditions of the oral environment, it is still relevant for prediction of clinical performance. Within the limitations of this *in vitro* study, the findings reject the research hypothesis, there doesn't suggest similar wear resistance and microhardness in the tested materials.

As previously described [17], composite abrasion occurs mainly in two steps. Initially, there is a selective wear in the organic matrix, which

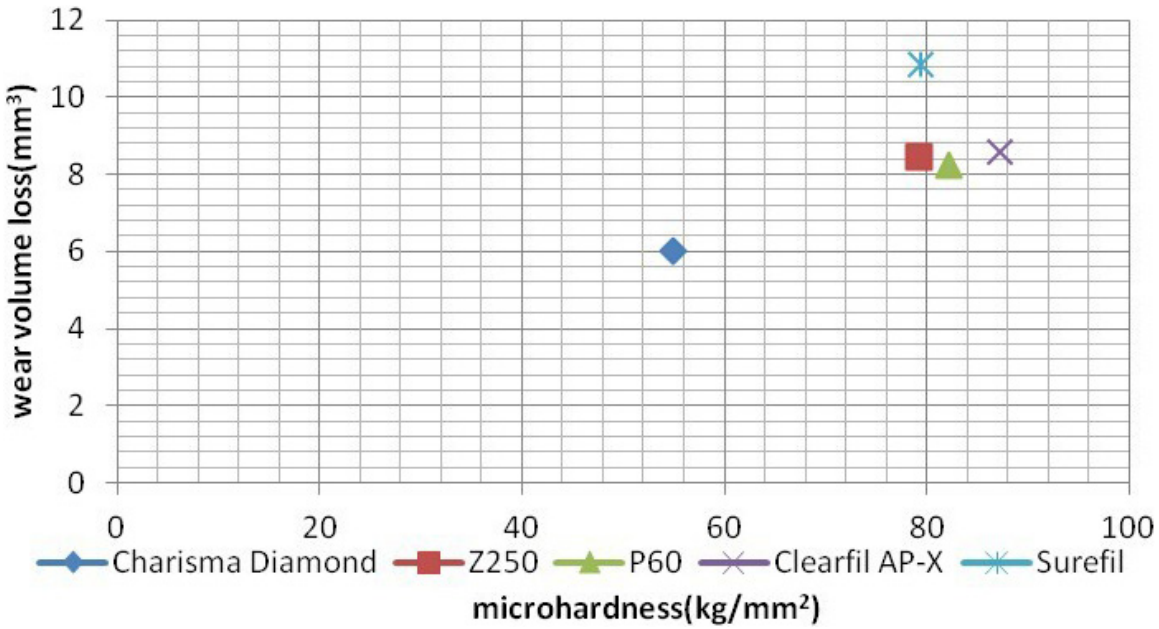

Figure 3. The relationship between wear volume loss and microhardness of experimental composites.

leads to exposure, protrusion and loss of inorganic particles. Afterwards, due to mechanical stress, these particles thus offer protection for the organic matrix, reducing its wear process [18].

Different methods have been employed to evaluate quantitative and qualitative abrasion resistance of composite resins. Teixeira *et al*. [19] measured the difference in specimen thickness from their initial thickness using a micrometer caliper. In many published studies on resin composite wear, only surface roughness parameter is reported [20, 21]. Such an approach has however serious shortcomings, since assessment of surface texture as a single parameter disregards materials such as Tetric EvoCeram or Grandio that showed low surface roughness and extremely high loss of substance or very little wear and quite high surface roughness in the study [22]. Determination of specimen weight loss after being subjected to toothbrush abrasion is another method used [23, 24]. This method has certain limitations when materials with high abrasion resistance and limited numbers of brushing strokes are investigated. Measurement of depth of wear with a computer-controlled three-dimensional measuring microscope and computing the volume loss from such data is proposed [25]. In addition, many scholars use a profilometric method to determine the depth of wear [22, 26]. This method is advantageous as wear depth and roughness of the worn surface are determined consecutively with the same instrument. Since it is a contact detection method, the scratches would be left on the specimen surface, which may affect the final results. With the emergence of the three-dimension non contact optical profilometer, the above-mentioned trouble could be solved. In agree with a previous study [27], we decided to use a three-dimension non contact optical profilometer to determine the wear volume loss.

In the present study, each composite resin presented a distinct performance, which suggests that results were dependent upon each formulation. It has been reported that the filler particles play a particular important role for both hardness and wear resistance. Condon [8] highlighted that the effect of filler volume on wear resistance follows a linear relationship, with high filler volumes decreasing wear rates due to the lower expanse of resin unprotected by filler particles, which was supported by other researcher [28]. For the composite resins investigated in this study, however, regression analysis showed no correlation between the wear resistance and hardness.

According to other literature [29], the filler content of composite material does not influence wear but other mechanical properties, such as diametral tensile strength and Knoop hardness. The weight fractions of filler particles of

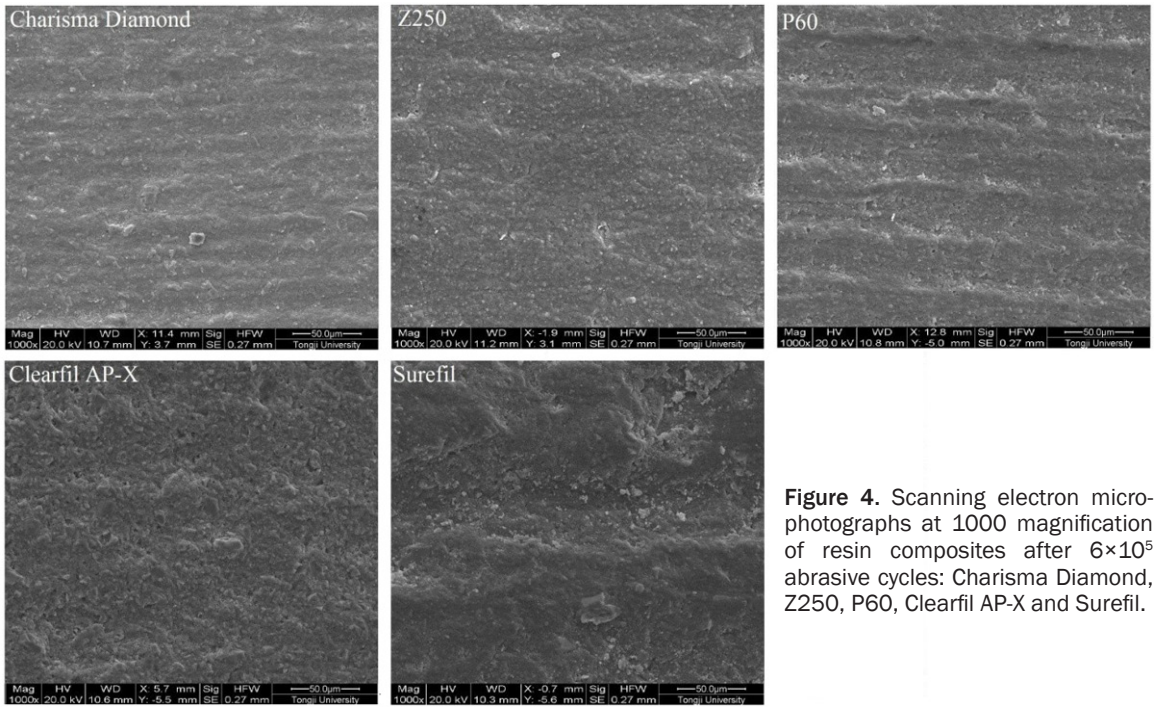

Figure 4. Scanning electron micrographs at 1000 magnification of resin composites after 6×10^5 abrasive cycles: Charisma Diamond, Z250, P60, Clearfil AP-X and Surefil.

the composites tested in this experiment ranged from 72 to 86wt%, and the Charisma Diamond with approximately 80wt% of filler content demonstrated the least W, although the filler content of the Clerafil AP-X was the greatest. The finding is accordance with Hu's [30] opinion that there is a threshold filler weight near 80% above which wear resistance is decreased.

The Clerafil AP-X showed higher W despite of its highest H and filler weight. As a microhybrid composite, the filler average size is larger than others in this study, which has been attributed to the fact that an increase in fill size causes an increase of the coefficient of friction, the contact forces and, thus insufficient wear resistance. Furthermore, Bayne *et al*. [31] suggested that the presence of large particles may theoretically cause greater wear of the restorative material. When the restoration is subjected to masticatory forces, the stress spreads through the filler particle into the resin matrix. This process results in the easy removal of these particles from the surface, thereby exposing the organic matrix and further accelerating wear.

Confirming the expectation that as particle size is decreased so is the wear [7], the conventional hybrid composite Z250 was found intermediate W and H. And a comparatively greater number of particles were probably present on the surface. Consequently, a larger contact area may have been established between the fillers and the counterpart, resulting in improved wear resistance.

One suggestion for improving the wear resistance of composites is to increase the abrasive resistance of the resin matrix, rather than increasing in the hardness of the filler particles [32, 33]. The predominant base monomer used in commercial dental composites has been bis-GMA, which due to its high viscosity is mixed with other dimethacrylates, such as TEGDMA. UDMA corresponds to another alternative organic matrix composition and it is often present in current compositions. Söderholm *et al*. [34] considered the urethane-base composites performed significantly better wear resistance than those which were bisGMA-based over three years clinical observation. It was contrary to this study which UDMA (Surefil) didn't show the well wear resistance. Furthermore, Kawai *et al*. [32] suggested that the TMPT-TEGDMA resin showed the most wear resistance, while Bis-GMA- and UDMA-based resins showed increased wear resistance with an increased content of TEGDMA. Different formulations are tried by manufacturers in an attempt to overcome the shortcomings; however, further inves-

tigations need to be performed to evaluate whether these changes promote superior mechanical properties.

Only little information is available about the TCD-DI-HEA monomer. Charisma Diamond is characterized by the presence of it, that according to the manufacturer combines low shrinkage with low viscosity and may account for the lower stress values recorded with this material. As for its wear resistance, almost no literature involved. It's only in Suzuki's [22] paper that it showed the higher wear resistance than other nanofill and nanohybrid. Of course, it shows the best wear resistance in this study.

Various studies have evaluated material surfaces by SEM after a wear process [17, 35]. O'Brien and Yee observed five principal wear standards of composite restorations: fracture, loss of particles of filler, wear of the resin matrix, failure of the matrix through cracking, and exposure of air bubbles [17]. These events were noticed in the present study (see **Figure 4**). SEM illustrations were important to determine the wear patter of the experiment composite resins.

Generally, it is desirable for any dental materials to yield wear behavior similar to the oral environment. So *in vivo* studies have been used cast replicas for the analysis of clinical composite wear, and found lower vertical substance loss for similar materials than observed *in vitro* study [36]. The similar results have been reported by Swift *et al*. [37], who found clinical wear rates lower than 100μm for the microfilled composites EsthetX and Point 4 in class I restorations after 36 months. The differences between the date gathered *in vivo* and *in vitro* might be due to the more complex wear *in vivo*, which may be considered as a result of the contact with different interfaces rather than a sole contact with an antagonist in a two-body wear assay [36]. Thus, a combination of at least two different wear settings has been proposed for the evaluation of wear behavior of dental restorative materials [38], which underlines the need for further studies dealing with the wear behavior of novel dental composites. Nevertheless, with the limitations of this *in vitro* study, the findings indicate the test resin composites yield the different wear behavior.

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