Wear Patterns of Adhesive Interfaces over Different Materials

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Abstract: *Purpose:* The aim of this *in vitro* study was to investigate differences in surface wear of enamel-material and dentin-material bonded interfaces obtained from upper molars samples and subjected to cycling occlusal load.

Methods: Forty-eight flat specimens of enamel-material and dentin-material bonded interfaces were prepared using different restorative materials with both CAD-CAM techniques and direct techniques. After the bonding and cementation procedures specimens were tested with a chewing machine with a stainless-steel ball on flat sliding contact (20N loads, 50.000 cycles). Wear analysis and comparison of the enamel or dentin substrates and the three restorative materials was performed using a 3D profilometer and analyzed with ANOVA test and post-hoc comparison procedures. Finally worn surfaces were examined with optical microscopy.

Results: Statistical analysis after simulated chewing cycles identified a significant influence of the factor "substrate" ($p<0.05$) and of the factor "restorative material" ($p<0.05$). The enamel results in being more wear resistant than dentin, and also more resistant than all the restorative materials tested. Considering the materials, the most severe wear loss was observed with micro-hybrid composite paste. CAD-CAM materials showed a wear rate significantly better both in association with enamel and dentin. An interesting wear pattern was found at the bonded interface level and the oval shapes obtained from the profilometer images underlined a repetitive wear pattern with the central zone more consumed and decreasing depth moving towards the perimeter. Initials signs of cracks were showed in enamel interfaces at the optical microscopy analysis.

Conclusions: Both the dental substrate and the restorative material significantly affect the wear behavior of a tooth-material interface after cyclic fatigue. Thus, the initial null hypotheses were rejected.

Keywords: Interface, Wear, Cyclic fatigue, CAD/CAM materials.

1. INTRODUCTION

In the perspective of a minimally invasive dentistry, the main goal is to maintain the maximum quote of dental tissue. In restorative dentistry it results in an increasing use of the adhesive techniques which provides the presence of an adhesive interface following the pioneer approach of Buonocore [1]. However, the integrity of the adhesive interface overtime is fundamental to increase the restoration longevity [2, 3]. Indeed, regardless the enormous advances in adhesive dentistry, the bonded interface stability still remains an issue. Among the different factors involved in marginal degradation, some are considered pivotal: insufficient resin impregnation of dentin, high permeability of the bonded interface, sub-optimal polymerization, phase separation and activation of endogenous collagenolytic enzymes [4] and differences in mechanical properties of tooth substrates and restorative materials.

Tooth wear is a multifactorial condition leading to the loss of dental hard tissues (enamel and dentin). It can be divided into mechanical (attrition and abrasion produced respectively by the tooth-to-tooth contact and the interaction between the teeth and other substances) and chemical wear (erosion produced by a chemical process) [5-7] and the restorative adhesive treatment represent a valid option to re-establish vertical dimension, occlusal pattern and aesthetic without removing sound enamel and dentin.

Resin based restorations are reliable materials that can be used in direct and indirect approaches, which showed a survival rate of 98% after 11.7 years and a failure rate of 2% [8]. The survival rates for resin inlays and overlays are between 92% and 95% at 5 years and 91% at 10 years and fractures are the most frequent cause of failure [9].

The literature suggests that direct composite techniques show less mechanical resistance compared with CAD-CAM indirect techniques due to a number of factors including the absence of post-polymerization, the major contraction, the great incidence of operator expertise, the different elastic modulus [10]. Indirect composite restorations showed superior clinical

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performances than direct composite restorations in spite of a greater loss of tooth structure and more clinical steps and fabrication procedures than direct restorations. However, several studies showed that there is no clinical significant difference between direct and indirect restorations in normal as well as in severely worn teeth [11, 12].

To date, there is a large variety of adhesive restorative materials such as ceramic, resin composites, CAD-CAM composites and hybrid ceramic materials, each one with their specific mechanical and wear properties. It is reported that volumetric wear of composite and resin-based materials are comparable (respectively until 102 µm and 225 µm per year), even if the composites are generally more resistant. As regard ceramics, zirconia and lithium disilicate have been reported to have a reduced quote of vertical wear (for a maximum of 116 µm, 127 µm and 148 µm in a year) than resins [5].

Enamel and dentin showed different behaviors regarding cyclic loading wear resistance. It is reported that under physiological pH conditions, dentin usually wears faster than enamel [13] because of its own structure and composition. Human tooth enamel exhibits a unique microstructure able to sustain repeated mechanical loading during dental function [14]. The fully formed (mature) enamel has unique morphological and biomechanical properties [15]. The mechanical parameters of human enamel, in particular Young's elastic modulus, ranges from 64.50 to 80.46 GPa [16], which is significantly different from dentin and it conducts to the conclusion that the two dental substrates are different in mechanical and tribological properties.

The mechanical properties of restorative materials have a direct influence on the performance of the whole tooth-restoration complex, affected by the stress distribution caused by masticatory loading [17]. The inconsistency between the tribological behavior of enamel and restorative material could be critical when their interface is directly placed in correspondence of an occlusal contact point [18]. In fact, according to Ferracane *et al.* [19], the cyclic occlusal contact on the tooth-material interface could cause an irregular wear, which could led to a marginal breakdown. Thus, despite the great evidence about the CAD-CAM materials properties and the evidence of differences in the wear mechanism of dentin and enamel substrate, there is still lack of knowledge regarding the wear of an adhesive interface when submitted to cyclic load. In this context, the aim of the present study was to evaluate the volumetric wear of the bonded interface when mechanically loaded. The null hypothesis tested were that the wear pattern involving the bonded interface is not influenced neither by the enamel and the dentin portion (1) neither by different restorative materials (2).

2. MATERIALS AND METHODS

2.1. Sample Preparation

24 intact upper molars, extracted for periodontal reasons, were selected and stored in sterile water at 37°C. After root debridement with ultrasonic devices, the samples were randomly divided into 3 groups (n=8 each) according to the restorative material employed (Table **1**):

- Group 1 (G1): CAD-CAM nano-hybrid composite (*Grandio Blocs, Voco GmbH, Germany)*
- Group 2 (G2): CAD-CAM hybrid resin (*Vita Enamic, Vita Zahnfabrik, Germany*)

Table 1: Composition of the Materials Employed in the Present Study

Material	Category	Manufacture	Composition	Production
Grandio Blocs	CAD-CAM Nano-hybrid composite	Voco GmbH	Nano-hybrid polymer inorganic fillers (86%) Polymer part: UDMA, DMA	Germany
Vita Enamic	CAD-CAM Hybrid-resin	Vita Zahnfabrik	Ceramic fillers (86%): SiO_2 (58-63%), Al_2O_3 (20-23%), Na_2O (9-11%), K_2O (4-6%), B_2O_3 (0.5-2%), ZrO_2 (< 1%), CaO (< 1%) Polymer part (14%): UDMA, TEGDMA	Germany
Venus Pearl	Micro-hybrid paste composite	Kulzer GmbH	2-Propenoic acid, 1,1'-[(octahydro-4,7-methano-1Hindene-5,-diyl)bis(methyleneoxycarbonylamino2,1-ethanediyl)] ester (10-25%), diuretan-methacrylate (5-10%), trietilen glicole dimethacrylate (1-5%), oxybenzone (0.25-1%)	Germany

- Group 3 (G3): micro-hybrid composite *(Venus Pearl, Kulzer GmbH, Germany)*

All samples were treated as follow: the roots were removed using a diamond saw with cooling water (*LS2, REMET, Italy*). The remaining crowns were divided in two halves along the mesio-distal sulcus obtaining one half of the crown with the buccal cusps and the other half with the oral cusps. For each group 16 half-crowns were obtained and randomly divided in 2 subgroups: 8 were used to create and evaluate adhesive interfaces between enamel and a restorative material (Subgroup A), the remaining 8 halves were prepared to evaluate the adhesive interface between dentin and the restorative material (Subgroup B).

Then, CAD-CAM blocks (size 14, shade A2 HT) of G1 and G2 materials were selected and sectioned in 3 mm thick slices with a diamond saw. For G3, an appropriate mold (dimensions: 14 mm x 14 mm) was created with polyvynilsiloxane *(Imprint II Garant Light Body, 3M ESPE, USA*) and employed to place 1mm thick layers of nano hybrid composite until reaching 3mm of thickness. The composite was cured for 60 seconds with a LED lamp (*Starlight Uno, MECTRON, Italy*) for every layer and then extracted from the mold [20].

2.2. Adhesive Procedures

The internal flat surface of each half tooth was etched with phosphoric acid gel (*35% phosphoric acid gel, Gel Etchant, Kerr, Switzerland)* for 30 seconds on enamel and 15 seconds on dentin, rinsed for 60 seconds with water and then air dried. A universal adhesive (*Adhese Universal VivaPen, Ivoclar Vivadent, Liechtenstein*) was applied over the etched surface following the manufacturer instructions.

On the other hand, the restorative material was treated as follow: G1 and G3 specimens were sandblasted with 50µm alumina particles for 5 second at 2 bar, rinsed with water for 30 sec and air dried. G2 specimens were etched using 9.6% hydrofluoric acid (*Porcelain etch gel, Pulpdent corporation, USA*) for 20 seconds, rinsed with water and air dried. All specimens were then ultrasonically cleaned (*VGT-800, Floureon, China)* for 5 minutes in alcohol and subsequently silanized with a ceramic silane (*Porcelain silane, BMJ Laboratories Ltd., Israel*). After evaporation at 100°C, over the pre-treated surface the universal adhesive (*Adhese Universal VivaPen*) was applied following the manufacturer instructions.

2.3. Luting Procedures

Every restorative material specimen was luted to the prepared tooth sample with a dual-curing resin cement (Bifix QM, Voco, Cuxhaven, Germany) with a thickness of 100 µm and then light-cured with a LED lamp (*Starlight Uno, MECTRON, Italy*) for 120 seconds for each side (occlusal, mesial, distal, vestibular). The interface width of all specimens in the present study was 100 µm measured with an adequate thickness sheet so this was all within the clinically acceptable range.

After 7 days of storage in sterile water at 37°C, the occlusal surface of all specimens was flattened using decreasing grit abrasive papers (#800, #1200, #2400, #4000) (*LS2, REMET, Italy)* until a smooth surface with a flattened enamel- (subgroup A) or dentin- (subgroup B) interfaced with the tested restorative material was obtained.

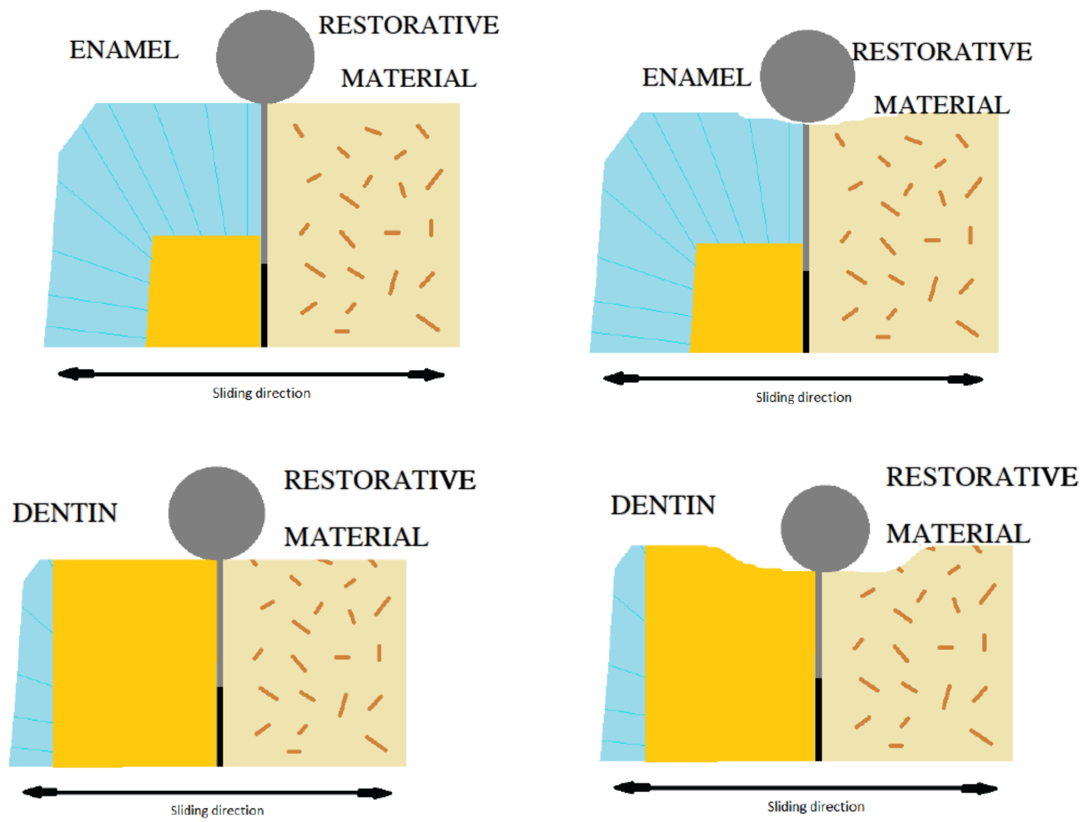
2.4. Wear Tests

A cyclic load was applied over the flat surface of each specimen using a mechanical chewing simulator (*Chewing Simulator CS-4, SD Mechatronik, Germany*) at room temperature using a ball-on-flat contact mode. The testing parameters included a contact load of 20 N, sliding rate of 8 mm/s, articulation frequency of 1 Hz, and displacement amplitude of 5 mm. The displacement midpoint was located at the bonded interface and the sliding movement started on the material side towards the teeth (Figure **1** and Figure **2**). The stylus alignment was ensured by the machine's function of alignment. The number of cycles was set at $5x10^4$ using a steatite ball with a diameter of 6.35 mm as antagonist, according to Arola *et al.* [2].

2.4.1. Wear Rate Assessment

After chewing simulation, all specimens were scanned with a 3D Laser scanner (*LS-LAS20, SD Mechatronik, Germany)* to calculate and observe the wear depth of the tracks. The resolution of the scan was set on 0.005 mm, with the smoothing function activated. The starting point, the end point and the mid-point of the wear tracks were manually set to border the worn area.

Three-dimensional topography maps of the wear area were reconstructed using the software (*Gwyddion 2.30, Department of Nanometrology, Czech Metrology Institute*) and different measures of wear depths were collected on the main chewing axis. For enamel and dentin surfaces, the resin cement and the restorative



materials, the maximum wear depths of the wear tracks were volumetrically measured as follow: a flat surface was created using three points outside the wear area of the sample. Moreover, linear profiles were obtained along the major axis of the wear track. The wear depth, calculated between the virtual flat surface and the linear profiles along the wear tracks, were calculated, starting from the luting cement interface as midpoint. Then, other measurements were performed to assess the tooth substrate (enamel or dentin) and the restorative materials loss at 100µm, 200µm, 300µm and 400µm from the cement midpoint.

2.5. Optical Microscopy

After wear tests, specimens were ultrasonically cleaned for 5 minutes in 90% ethanol. After that the samples were observed with the optical microscope *(Axiocam 208 color, Zeiss, Turkey)* to obtain detailed images of the surface damages after the fatigue test and analyze the differences at 60x.

2.6. Data Analysis

Since data were normally distributed, all the obtained results were statistically analyzed with a two-way ANOVA test and a Tuckey post-hoc test with Bonferroni correction. Statistical analysis was performed with a dedicated software (Stata v.14, StataCorp). Significance was set for $p \leq 0.05$.

3. RESULTS

Wear tracks appeared in all specimens with a conventional oval-shape, with the deepest area located in the central region. This profile was obtained with a section along the major axis of the wear pattern (Figure **3** and Figure **4**).

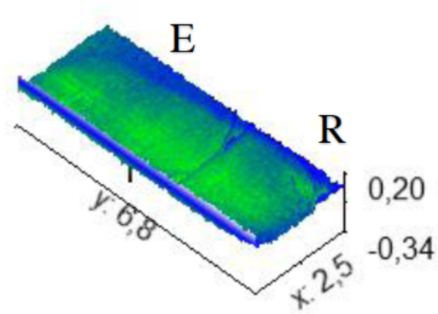


Figure 3: Example of wear pattern of an enamel-material specimen, showing the oval shape of the wear track, the interface and the major depth at the interface. E: the enamel, RM: the resin material. Note the distinct transition in depth of wear from the enamel to the bonded interface and resin composite.

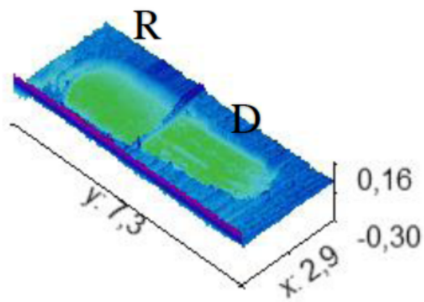


Figure 4: Example of wear pattern obtained from 3D images obtained with the laser profilometer, showing the oval shape of the track, the interface, the major depth at the interface. D: the dentin, RM: the resin material. Note the distinct transition in depth of wear from the enamel to the bonded interface and resin composite.

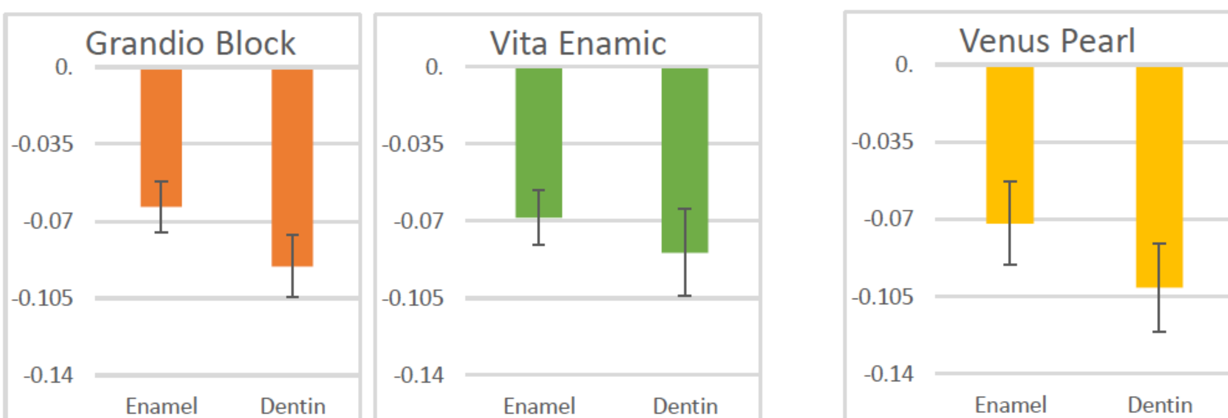
The mean wear rate of enamel and dentin substrate, expressed in mm ± standard deviation, after cyclic fatigue over an adhesive interface with different restorative materials are reported in Table **2** and Graph **1**.

ANOVA test showed that the tooth substrate significantly influenced the wear rate (p<0.05), whit enamel performing significantly better than dentin. No significant interactions were reported among other variables; therefore, it can be concluded that the mean wear of enamel is inferior compared to the wear of dentin independently on which material they are associated with.

The mean wear rate of the restorative materials, expressed in mm ± standard deviation, after cyclic fatigue over an adhesive interface with enamel and dentin are reported in Table **3** and Graph **2.**

Table 2: Mean ± Standard Deviation, Expressed in mm, of the Wear rate of Enamel and Dentin

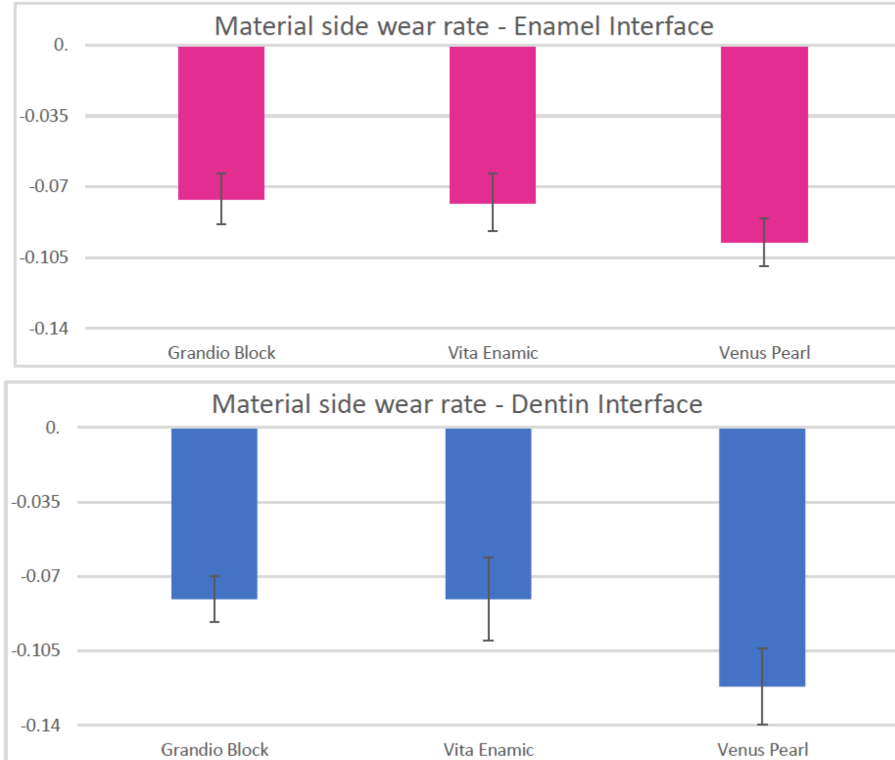
	Grandio Block	Vita Enamic	Venus Pearl
Enamel	-0.063 ± 0.0232	-0.068 ± 0.0247	-0.072 ± 0.0375
Dentin	-0.091 ± 0.0282	-0.084 ± 0.0394	-0.101 ± 0.0310



Graph 1: A graphical representation of the wear rate of enamel and dentin after cyclic fatigue towards an adhesive interface enamel and dentin.

Table 3: Mean ± Standard Deviation, Expressed as mm, of the Wear Rate of Tested Restorative Materials

	Enamel	Dentin
Grandio Block	-0.0761 ± 0.0251	-0.0808 ± 0.0217
Vita Enamic	-0.0777 ± 0.0284	-0.0807 ± 0.0389
Venus Pearl	-0.0974 ± 0.0238	-0.1218 ± 0.0177



Graph 2: Graphical representation, expressed in mm of the wear rate of restorative materials after cyclic fatigue towards an adhesive interface enamel and dentin.

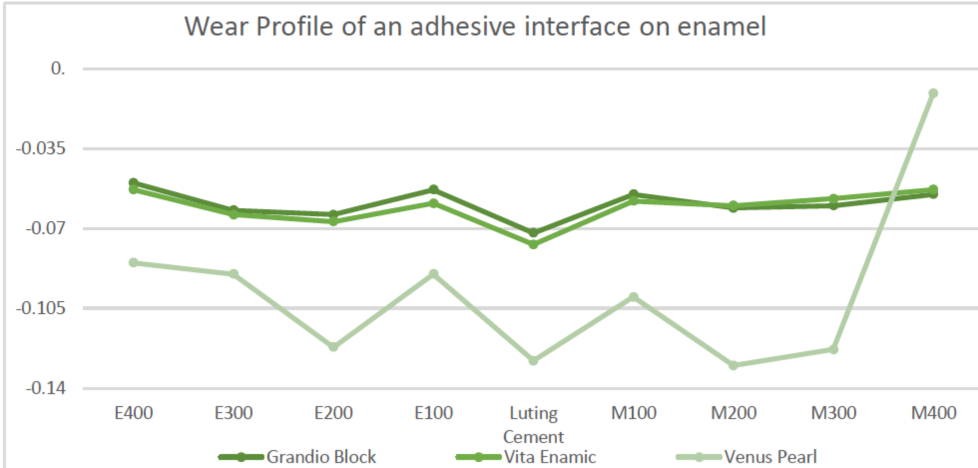
Representative wear profiles obtained in subgroup A and subgroup B are displayed in Graph **3** and **4** respectively.

The statistical analysis showed a significant influence of the restorative material tested on the wear rate close to the luting cement interface. The post-hoc analysis showed that mean wear of nano-hybrid composite is significantly higher than CAD-CAM nano-hybrid composite and CAD-CAM hybrid ceramic, independently on which substrate they are associated with. Moreover, the association of enamel or dentin with the three different restorative materials has shown no significance influence of the wear rate.

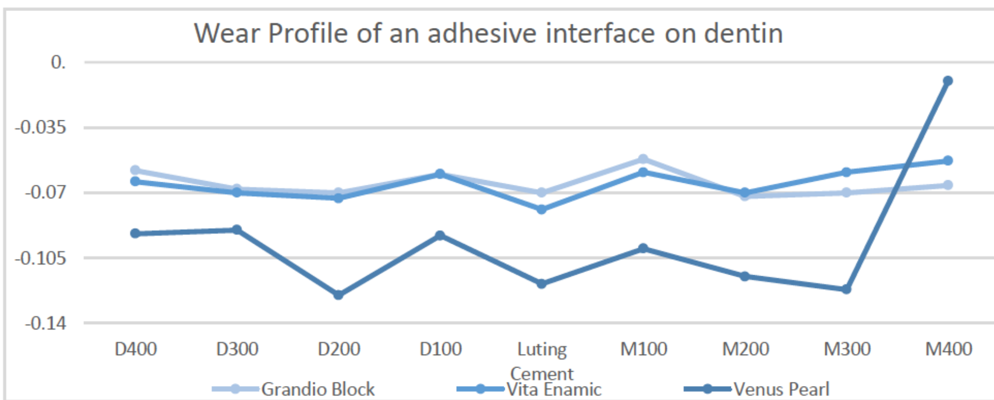
4. DISCUSSION

The present *in vitro* study was realized to evaluate the wear resistance of an adhesive interface created between tooth substrates and modern restorative materials. According to the obtained results, the first null hypothesis was rejected since enamel showed significantly lower wear compared to dentin close to the luting cement layer. As reported in literature, the two substrates are really different in terms of anatomical structure and properties [21-23], thus leading to a different tribological behavior. The principal microstructural feature of enamel are the prisms, which are approximately 5 to 8 μm in diameter and consist of an assembly of needle-like apatite crystals that are consolidated into regular prism shapes by a very thin layer of enamel proteins [2]. From a structural point of view, enamel prisms are highly mineralized, with approximately 70%-98% vol mineral, and the remaining part consisting of organic proteins and water [24]. Near the surface of the tooth the prisms extend towards the dentin-enamel junction as a series of parallel columns. However, in the inner enamel the prisms are “decussated”, which means crossed in an X form, into a pattern which has the interesting capacity for conferring crack growth resistance [25]. On the other hand, the structure of dentin consists of 80% hydroxyapatite and 20% of organic material, which is the reason why its mechanical and responsive capacities are different from enamel.

Concerning the second null hypothesis, significant differences in wear rate were highlighted between CAD-CAM materials tested and a nano hybrid composite. Recently, polymer-infiltrated network ceramics, such as Vita Enamic, composite nanoceramics, such as Grandio Block, have been introduced. Vita Enamic is composed of a porous ceramic network (86%), which is then infiltrated with a



Graph 3: Comparison between the wear profiles obtained at the luting cement interface (midpoint) and at 100µm, 200 µm, 300 µm, 400 µm at both enamel and restorative material sides. E: the enamel, M: restorative material. It is evident the minimal wear of the enamel side and the great difference in wear rate between CAD-CAM materials and the nano hybrid composite. Note the profile obtained before and after the luting cement midpoint, where the lowest peaks are located.



Graph 4: Comparison between the wear profiles obtained at the luting cement interface (midpoint) and at 100µm, 200 µm, 300 µm, 400 µm at both dentin and restorative material sides. D: the dentin, M: the restorative material. The greatest wear rate is located at the dentin side and a difference between CAD-CAM materials and nano hybrid composite is also observed.

polymer by capillary action, while composite resin nanoceramic blocks consist of a polymeric matrix reinforced by nanohybrid fillers. Industrial fabrication of these blocks under high temperature and high pressure has led to a higher volume fraction filler and higher conversion rates (85%) than layered composites for direct restorations, thus significantly improving their mechanical properties [26, 27]: flexural strength is from 105 MPa to 219 MPa, flexural modulus is from 8 GPa to 32 GPa, the resilience modulus is from 0.21 MPa to 3.07 MPa [28]. Consequently to those properties and characteristics, all CAD-CAM restorative materials, and in particular the two evaluated in the present study, exhibited high values of fracture and flexural resistance, making them suitable materials for posterior restorations in worn dentition [29].

On the other hand, composite materials for direct restorations also showed good mechanical and tribological properties to occlusal loading which makes them suitable for posterior restoration, even if some criticism could be observed. The mean degree of conversion varies between 53% to 79% [30] and the polymeric structure of direct composites is not always homogeneous after light curing direction [30]. Moreover, nanofilled composite exhibited lower flexural strength and less favorable elasticity modulus than CAD-CAM materials [31]. Under sliding contact with the ceramic ball, the polymer matrix of direct resin composite undergoes deformation and wear more than the tooth substrate and the hybrid cement layer, which exposes the fillers and enables their release [2]. For all those reasons an important difference in terms of wear is reported between direct composites and indirect CAD-CAM resin-based materials. On the other hand, no significant differences were reported between the two CAD-CAM materials. This result is probably due to the similarities in their structure which make them quite comparable in terms of wear-resistance properties [32].

It is reasonable to assume that an ideal restorative material should have mechanical and tribological properties similar to the dental tissues [2, 33]. Previous researches reported that enamel is more wear-resistant than resin composite under the same experimental conditions [2, 34, 35]. However the materials tested exhibited significantly lower resistance to wear than the enamel but not than the dentin substrate. The divergence of the moduli between the restorative materials and enamel could cause a contact stress concentration between the enamel and the adjacent bonded interface [36], which could influence the wear behavior at the cement interface where the forces are significant and the response of the enamel is very strong in comparison with the restorative materials [37].

In conclusion, according to the obtained data, it can be stated that enamel substrate is more resistant to cyclic loading forces than dentin and other tested restorative materials. Moreover, CAD-CAM resin-based materials tested showed a better tribological behavior compared to direct nano-hybrid composite both with dentin and enamel substrates. Thus, it could be suggested to avoid the placement of an adhesive interface in correspondence of a sliding occlusal contact point when occlusal enamel is preserved. However further studies are necessary to better investigate the topic and simulate different clinical conditions, also taking into account the limitations of present study.

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