Two-body wear simulation influence on some direct and indirect dental resin biocomposites - A qualitative analysis

ADRIANA CARACOSTEA¹*, NADINA MORAR², ADRIAN FLOREA³, ANDRADA SOANCA⁴, MINDRA EUGENIA BADEA⁵

Purpose: The aim of this study was to qualitatively assess the outcomes of two in vitro aging methods, thermal-cycling and two-body wear simulation accomplished with a dual-axis chewing device, on the surface characteristics of eight direct and indirect dental resin biocomposites. Methods: Eighty mesial-occlusal-distal dental cavities were restored with four direct nanohybrid composite materials and with four nano- and micro-hybrid lab-fabricated resin composite inlays. After the restored teeth were subjected to thermal-cycling and wear simulation based on mechanical loading, the surface texture features of the restorations were separately analysed for each of the methods, on epoxy resin models using a digital camera, computer-aided-design system, optical stereo-microscopy and scanning electron microscopy. Results: All the dental restorative resin based composites used in this investigation displayed different cyclic wear patterns after undergoing mechanical loading. After thermal-cycling, the group of resin composite inlays showed a better adaptation, a smoother and more polished occlusal surface compared with direct restorative materials. Only two of direct nanohybrid resin composites performed better after two aging methods. One nanohybrid and the other two microhybrid resin inlays did not perform as expected when they were subjected to simulated wear compared to the rest of test materials. Conclusions: The use of the two-body wear simulation method revealed important information about the behavior of the dental resin-based composites when multiple oral factors are involved in a lab-simulated condition. Furthermore, the macro- and micro-morphological analysis showed different abrasion patterns among the materials being tested according to the filler percentage and distribution of the particles within the resin matrix.

Key words: scanning electron microscopy, optical microscopy, two-body wear, CAD system, advanced biocomposites

1. Introduction

Reconstruction of missing hard dental tissues has always been an important issue for operative dentistry. Thus, new types of direct and indirect resin composite biomaterials are developed to restore especially the posterior teeth, where occlusal forces have a higher impact on this area (20–120 N) [12]–[14]. Factors such as occlusal wear, temperature changes and mechanical loads may influence

their behaviour until the loss of the whole dental restoration [12].

Fatigue and wear represent a complex phenomenon with a variety of factors which affect both natural teeth and biomaterials used to restore them, especially when wide posterior stress-bearing restorations are involved [12], [13], [23]. Reported values of clinical wear rate for posterior teeth differ from one study to another. Thus, in the past years, researchers have tried to develop new experimental methods and protocols used to simulate wear or functional masticatory processes in

Received: October 10th, 2015

Accepted for publication: December 12th, 2015

Department of Dental Materials and Ergonomics, "Iuliu Hatieganu" University of Medicine and Pharmacy, Cluj-Napoca, Romania.
² CAD-CAM Center, Private dental laboratory, Cluj-Napoca, Romania.

Department of Cell and Molecular Biology, "Iuliu Hatieganu" University of Medicine and Pharmacy, Cluj-Napoca, Romania.
 Department of Periodontology, "Iuliu Hatieganu" University of Medicine and Pharmacy, Cluj-Napoca, Romania.
 Department of Preventive Dentistry, "Iuliu Hatieganu" University of Medicine and Pharmacy, Cluj-Napoca, Romania.

^{*} Corresponding author: Adriana Caracostea, Department of Dental Materials and Ergonomics, "Iuliu Hatieganu" University of Medicine and Pharmacy, Cluj-Napoca, Romania, 15 Babes Street, 2nd floor, Cluj-Napoca, Romania. Tel: +40745295479, e-mail: adriana.caracostea@umfcluj.ro

the lab, for a better prediction of the clinical phenomenon [1], [11], [18], [22], [23]. Starting from 2001 a technical ISO specification was presented, thus, based on these attempts for standardization of the two- and three-body wear tests, new devices have been developed several years later, trying to simulate oral movements. Such a medical device used in the past years, for in vitro aging tests, is the chewing simulator (Chewing simulator, CS-4.2, SD Mechatronik, Germany) [4], [8], [22]. It has been shown that CS-4.2 simulator leads to reliable results and provides an easier adjustment by applying simultaneously same number of weights for each test-chamber and the samples may also be thermocycled during mechanical movements, based on the software controlled by the operator [22].

Two-body wear may develop as a form of "non-masticatory motion" found usually in bruxism or as an attrition phenomenon, considered a "physiological balanced occlusion" [11] which occurs, especially, at the occlusal contact areas (OCA) [10], [11].

The loss of hard dental tissues involved in a mesial-occlusal-distal cavity (MOD) after removal of a decay, can affect the resistance of the tooth during masticatory processes or tooth-to-tooth motions at the occlusal contact areas of physiological occlusion [10]-[12], [14]. This type of cavity may be restored in two different ways: using direct resin-based composites (RBCs) or a lab fabricated restoration (i.e., resinbased composite inlays). For an indirect inlay one may use dental ceramics, resin composite, alloys, etc. Usually, due to financial problems or other reasons, the patient may request a direct MOD restoration, although, the use of indirect restorative materials is considered more advanced and indicated for restoring these types of stress-bearing cavities. In a given situation, the use of lab manufactured RBC inlays will assure and bring to the future restoration a material with higher mechanical and physical properties [21]. New properties and formulae improvements of the nano-filled dental biomaterials indicated for restoration of posterior teeth have lead them closer to the properties of natural dental tissues, increasing their possibility to resist the tribological mechanisms. Nevertheless, the material loss at the occlusal contacts during functional masticatory movements, is still considered the most important element of wear for these restorative biocomposites [12], [14], [23], [25].

The possibility to evaluate the surface texture details status of restorations over the years may be achieved based on the clinical trials [4], [14] or on oral environment-simulated in vitro methods [10], [18], [22], [23], [25]. For both situations, the researchers may use several well-known qualitative and quantita-

tive procedures: SEM, OM, restoration volume/height loss, etc. Mostly, when a dental restorative material is analysed, not only the quantitative methods count, but also a qualitative assessment might be used to give the dental practitioner a general overview.

Among all the micro-morphological methods, scanning electron microscopy (SEM) characterisation details may be used to offer information about surface micro-morphology, filler particles type, distribution and amount within the composition of different restorative materials [4], [20], [21]. However, this method has certain limitations, such as "artefacts", "loss of the surface details", "small defects of the sample after vacuum and high temperature exposure" [4]. Thus, in the past years, new techniques of analysing the micro- and macro-surface morphology have been developed, such as optical microscopy used with high magnification in combination with SEM evaluation [4].

Little information may be found in the Medline database regarding the use of high magnification optical stereo-microscopy, digital cameras and 3D laser scanner of computer-aided-design (CAD) systems, for in vitro macro- and micro-morphological surface assessment.

This study continues previous research in the field [15], [20], [21] aiming with this in vitro analysis to emphasize the importance of different qualitative evaluation methods (digital camera, optical microscopy, scanning electron microscopy and CAD system) of surface morphology and wear characteristics of four nanohybrid direct resin-based composites, and four lab fabricated composite inlays (two nanohybrid filled and two microhybrid filled, respectively), when subjected to two in vitro aging methods: thermal-cycling (TC) and simulated wear based on mechanical loading (ML).

2. Materials and methods

Eighty permanent third molars, free of fissures, cracks or caries, extracted for therapeutic reasons based on a written approved consent, were kept in 1% chloramine solution for 14 days and then in distilled water at 4 °C for maximum 60 days prior to their preparation. The soft tissues and calculus were removed with manual scalers. Standardized mesial-occlusal-distal (MOD) cavities were prepared using a diamond bur mounted on a water-cooled turbine. The inner surface of the cavities was smoothed with fine coarse cylindrical burs and no bevels were prepared at the margins.

Clinical use	Material/ Group	Filler type and weight percentage*	Organic matrix*	Composition*	Brand
Direct restorative materials	Premise(P)/ Group1	– Nano-hybrid – 84 wt.%	– Bis-GMA – TEGDMA	Pre-polymerised filler(PPF)Point 4 fillersSilica nanofiller	Kerr Corp., USA
	Venus Pearl(VP)/ Group 2	– Nano-hybrid – 80 wt.%	– UDMA – TCD-DI-HEA	Ba-Al-F glassPre-polymerised fillerSilica nanofiller	Heraeus Kulzer, Germany
	Kalore(K)/ Group 3	– Nano-hybrid – 82 wt.%	– UDMA – DX-511 monomer (DuPont monomer)	 Fluoro-alumino-silicate glass Strontium glass High density radio-opaque (HDR) pre-polymerized fillers Silica fillers 	GC Corp., America
	Beautifil II(BF)/ Group 4	 Nano-hybrid Giomer with fluoride release and recharge 83.3 wt.% 	– Bis-GMA – TEGDMA	Multifunctional glass fillers Surface Pre-Reacted Glass-ionomer(S-PRG) filler based on fluroboroaluminosilicate glass	Shofu Inc., Japan
Lab manufactured restorative materials	Premise Indirect(PI)/ Group 5	– Nano-hybrid – 84 wt.%	– Bis-GMA – TEGDMA	Pre-polymerised filler(PPF)Point 4 fillersSilica nanofiller	Kerr Corp., USA
	Signum Ceramis(SC)/ Group 6	Nano-hybrid73 wt.%	 Multi-functional methacrylic acid esters 	Glass-ceramic filler,Nanoparticles,Silica fillers	Heraeus Kulzer, Germany
	Gradia(G)/ Group 7	– Micro-hybrid – 75wt.%	– UDMA	Fluoro-alumino-silicate glassPre-polymerized fillerSilica fillers	GC Corp., America
	Ceramage(C)/ Group 8	– Microhybrid – >73 wt.%	– UDMA	Progressive FineStructured fillers (PFS)Zirconium silicate	Shofu Inc., Japan

Table 1. List of dental materials under study, their composition and filler type

BisGMA = Bis-glycidylmethacrylate; TEGDMA = Triethylglycidylmethacrylate; UDMA = Urethane dimethacrylate; TCD-DI-HEA = 2-propenoic acid, (octahydro-4,7 methano-1H-indene-5-diyl) bis(methyleneiminocarbonyloxy-2,1-ethanediyl)ester; DX-511 = high molecular weight polyurethane dimethacrylate monomer.

The teeth were randomly divided in 8 groups (n = 10 samples per each group). The standardized MOD cavities were incrementally restored (2 mm/layer) with the following nanohybrid direct resin composite materials: Group 1 – Premise (P) (Kerr Corp, USA); Group 2 - Venus Pearl (VP) (Heraeus Kulzer, Germany), Group 3 - Kalore (K) (GC Corp, America) and Group 4 – Beautifil II (BF) (Shofu Inc., Japan) (Table 1). Each of the materials was applied and bonded to the dental structures according to the manufacturer's recommendations [2], [9], [16], [24]. For light-curing of each increment a halogen lamp was used (Optilux 501, Kerr Corp, USA) (output >800 mW/cm²). The restorations were finished with multiple blade burs and fine coarse diamond burs and polished with abrasive discs and polishers (Optidisc, Kerr Corp., USA).

For the composite inlays a clinical protocol was followed according to reference [15] and the samples

were restored with the following indirect resin composites: Group 5 – Premise Indirect (PI) (Kerr Corp., USA), Group 6 – Signum Ceramis (SC) (Heraeus Kulzer, Germany), Group 7 – Gradia (G) (GC Corp, America), and Group 8 – Ceramage (C) (Shofu Inc., Japan) (Table 1). The lab fabricated composite inlays were luted with a dual-cured resin cement according to the manufacturers' recommendations [3], [6], [17], [19]. Removal of excess luting agent was done after 5 seconds of polymerisation. Additional 60 seconds of light-curing per each sample surface was done for the final setting of the resin luting cement. The set luting agent was then finished and polished with fine coarse discs and polishers (Optidisc, Kerr Corp., USA).

All the samples analysed were kept in distilled water at 4 °C prior to in vitro testing. After 30 days the restored teeth from all the groups were subjected to 1000 thermal cycles (TC) between 5–55 °C, a dwell time of 30 seconds and a transfer rate of 5 seconds.

^{*} According to the manufacturers' information [2], [3], [5], [8], [15], [16], [19], [24].

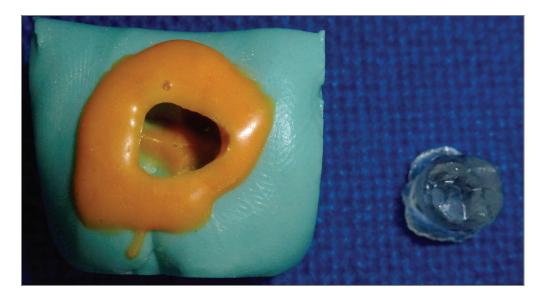


Fig. 1. Elastomeric impression and epoxy resin replica of the restored tooth

This corresponds to approximately 2 months of clinical performance [1]. After TC method had been finished, impressions of the restored teeth were taken with a condensation-cured silicone (Zetaplus Putty and Oranwash Light/ Zhermack, Italy) and poured with transparent epoxy resin (Epo-Kwick, Buehler Ltd., USA) to obtain the first set of replicas for SEM and the other methods (Fig. 1).

For the mechanical loading (ML), a dual-axis chewing simulator (Chewing simulator, CS-4.2, SD Mechatronik, Germany) was used. The teeth were embedded in self-cured acrylic resin (Duracryl /SpofaDental, Kerr Company) up to 2 mm from cemento-enamel junction and then mounted in the testchambers without any lateral inclination (Fig. 2). In this simulated setup, the steatite antagonist stylus had an axial direction towards one of the cuspal slopes. There was imagined a two-body wear scenario using distilled water as lubricant medium. Thus, with the provided software we were able to establish the parameters of the device as follows: 100000 unidirectional load cycles at 5 kgf (49 N) for each stylus barholder, at a frequency of 1.6 Hz and a lateral movement of 0.7 mm. During mechanical movements the test-chambers were filled with distilled water at room temperature, so that the samples should be fully immersed. The number of loaded cycles corresponds to 5 months of clinical service of the restorations in the oral cavity [1], [8], [11], [22]. After ML test had been completed, impressions of the restored teeth were taken again using the same type of impression material and a second set of transparent epoxy resin replicas was obtained (Fig. 1). The chewing simulation was performed at the Department of Dental Materials and Ergonomics, Faculty of Dental Medicine, University of Medicine and Pharmacy "Iuliu Hatieganu", Cluj-Napoca, Romania.

The specimens of each test material for both aging methods were assessed for qualitative analysis. Firstly, all the epoxy resin samples were sputtered-coated with a very fine layer of gold.



Fig. 2. Restored tooth mounted in the sample container of the dual-axis chewing simulator

The micro-surface analysis for both in vitro aging methods (TC and ML, was done using scanning electron microscopy (SEM) (Jeol, JSM, 25S, Jeol Japan) at different magnifications (45×, 450×) and optical microscopy (OM) with a light-inverted stereomicroscope (Olympus KC301, Olympus America Inc.) at 4× and 10× magnification. The surface details were analysed with the software provided (Quick-Photo Micro 2.3, Olympus America Inc.).

The macro-surface characteristics of the occlusal surfaces and the cuspal slope of the restorations involved were captured with a digital camera (DC) (Panasonic Lumix, DMC-TZ20, Japan) using 2.5× magnification and with a 3D dental lab laser scanner (D250 model by 3shape) in combination with CAD software (Dental SystemTM 2014 by 3shape, version: 2.9.9.3, WIELAND, Germany).

3. Results

A qualitative analysis of all the epoxy resin replicas of the resin-based composites studied was made according to three main criteria for macromorphological evaluation (corresponding features of surface details based on clinical elements for both restoration methods) and two main criteria for micromorphological analysis (corresponding features of surface details based on structure of the biocomposite materials tested), respectively, as described in Table 2. It is important to mention that during and after the in vitro aging methods were accomplished none of the teeth lost its restoration.

The CAD and DC analysis, after TC procedure, showed for the P restorations (Group 1) some irregularities of roughen occlusal surface and a limited opening at the tooth-restoration interface (Fig. 3A1, B1). After the material was subjected to mechanical loading (ML) one could observe that the P material had maintained a similar occlusal surface shape and surface polishability, but less along the cuspal slope where a V-shaped wear trace of the stylus can be ob-

Table 2. Criteria and features of the macro- and micro-morphological analysis				
used to evaluate the dental materials after in vitro aging methods				

Analysis	Criteria	Features
	Occlusal anatomical	– overhangs
	shape	– restoration contour
A. Macro-morphological	Marginal adaptation	 restoration-tooth interface integrity
analysis*	2. Marginar adaptation	 ledge formation
	2 S	 abnormal irregularities
	3. Surface polishability	surface roughness
	1.6.6	surface porosity
		 exposure of particles and resin
D.M. 1.1.1.1	Surface structure details	matrix
B. Micro-morphological	details	 debonding of the filler particles
analysis**		from resin matrix
	2. Fatigue-wear	– wear traces
	behaviour	– micro-cracks/fissures

^{*} Criteria and features analysed according to surface details based on clinical elements for both restoration methods.

^{**} Criteria and features analysed according to surface details based on structure and composition of the test materials.

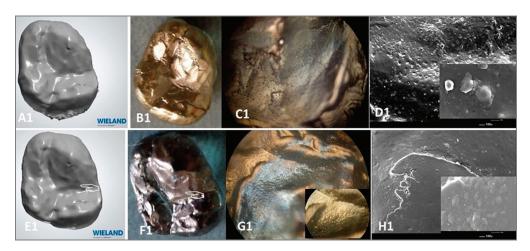


Fig. 3. Macro- and micro-surface analysis images for P samples (Group 1) (left to right: CAD, DC, OM, SEM) after TC (A1–D1) and after ML (E1–H1). DC magnification at 2.5×, OM magnification at 4× and 10× and SEM magnification at 45× and 450×

served (Fig. 3E1, F1). The micro-surface analysis (OM and SEM) indicated more structural details: surface porosity with round-shaped clusters and particles' exposure (Fig. 3C1,D1). The micro-morphological images of the teeth being tested after ML presented more exposed spherical clusters and rupture of the resin matrix with fillers than the images taken after TC method (Fig. 3C1, D1, G1, H1).

When macro-morphological evaluation after TC method was accomplished for VP samples (Group 2), the following observations were made: slightly porous occlusal surface and similar macro-percolation at the tooth-restoration interface compared with P samples (Fig. 3A1, B1, Fig. 4A2, B2). Comparatively, the CAD and DC images indicate same surface irregularities and a longer wear zone on the cuspal slope of VP restorations subjected to ML procedure (Fig. 4E2, F2). The OM method did not show any surface changes for the VP restorations, while the SEM images revealed a porous surface and pres-

ence of micro-fissures around the fillers (Fig. 4C2, D2). The OM and SEM observations could indicate micro-wear traces, micro-fissures within the material, rupture of resin matrix and exposure of filler particles (Fig. 4G2–H2).

The macro-morphological observations of the K specimens (Group 3) revealed marginal overhangs, some interfacial defects and a low degree of polishability (Fig. 5A3,B3). Moreover, a wider wear trace surface, ledge formation around the restoration contour and a lower degree of surface polish were observed after the material had been subjected to ML test (Fig. 5E3,F3). The microscopic image at 4× magnification (Fig. 5C3) presents an irregular surface with some occlusal defects, while the SEM analysis provides important information: a roughen surface with exposure of pre-polymerised fillers and with some defects around them (Fig. 5 D3). In addition, the analysis of Fig. 5G3 and Fig. 5H3 showed surface roughness with micro-fissures within the material,

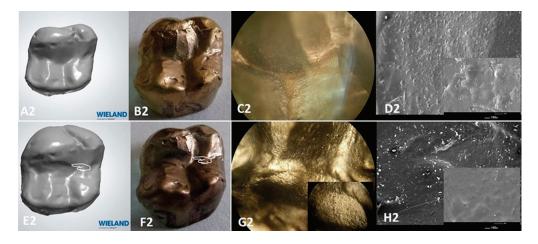


Fig. 4. Macro- and micro-surface analysis images for VP samples (Group 2) (left to right: CAD, DC, OM, SEM) after TC (A2–D2) and after ML (E2–H2). DC magnification at 2.5×, OM magnification at 4× and 10× and SEM magnification at 45× and 450×

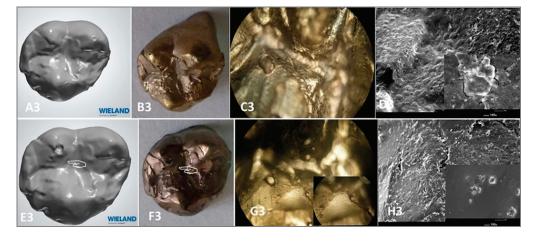


Fig. 5. Macro- and micro-surface analysis images for K samples (Group 3) (left to right: CAD, DC, OM, SEM) after TC (A3–D3) and after ML (E3–H3). DC magnification at 2.5×, OM magnification at 4× and 10× and SEM magnification at 45× and 450×

exposure of the filler particles and the presence of the same type of defects, as mentioned previously.

From a macro-evaluation point of view, similar surface characteristics between BF (Group 4) and K samples may be observed with the CAD and DC methods for both aging procedures (Fig. 5A3, B3, E3, F3, Fig. 6A4, B4, E4, F4). In addition, macroscopic evaluation methods showed a wider and deeper opening of the resin composite-dental tissue interface and an irregular and defective occlusal surface of the BF restorations compared with the other materials studied (Fig. 6A4, B4, E4, F4). The OM and SEM micrographs, after TC procedure, illustrate surface roughening, exposure of the fillers and micro-fissures within the material (Fig. 6C4, D4). In addition to the above-mentioned micro-morphological features, fatigue wear traces with filler particles and resin matrix dislocation were observed after the material had been subjected to mechanical loading (Fig. 6E4, F4).

Subjected to thermal cycling, the PI group showed at a macroscopic level (CAD, DC) no interfacial gap between the inlay and dental structure and reduced surface irregularities (Fig. 7A5, B5). When the samples of the same group were subjected to mechanical loading, a short V-shaped wear trace and a slight increase of surface porosity could be observed (Fig. 7E5, F5). The OM and SEM images revealed a smooth surface and a homogeneous distribution of the filler particles for the PI restorations (Fig. 7C5,D5). Exposure of the filler particles could be seen at 4× magnification of optical microscope (Fig. 7G5) and compact fracture-ledges of resin matrix with incorporated fillers observed in SEM images at 450× magnification (Fig. 7H5).

The results for SC group of the CAD and DC evaluation after TC procedure showed an opening of tooth-inlay interface, but with maintenance of the occlusal anatomical shape (Fig. 8A6, B6). The ML method revealed important occlusal morphology change of the cuspal slopes and macro-wear traces compared with the tested direct resin composites and PI samples (Fig. 8E6, F6). The OM method showed a porous surface of the composite inlay, while in the SEM micrographs, an exposure of more than two

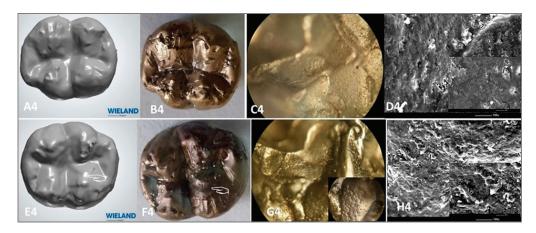


Fig. 6. Macro- and micro-surface analysis images for BF samples (Group 4) (left to right: CAD, DC, OM, SEM) after TC (A4–D4) and after ML (E4–H4). DC magnification at 2.5×, OM magnification at 4× and 10× and SEM magnification at 45× and 450×



Fig. 7. Macro- and micro-surface analysis images for PI samples (Group 5) (left to right: CAD, DC, OM, SEM) after TC (A5–D5) and after ML (E5–H5). DC magnification at 2.5×, OM magnification at 4× and 10× and SEM magnification at 45× and 450×

thirds of the filler particles could be seen (Fig. 8C6, D6). The results for ML method evaluated with SEM analysis at 45× and 450× magnification showed multiple micro-cracks and fissures within the material along and perpendicular to the wear direction, loss of

resin matrix of the worn mechanically loaded site and exposure and dislocation of the fillers (Fig. 8H6).

For G samples (Group 7), the thermal cycling aging method led to a lower degree of polishability, but without any surface contour changes (Fig. 9A7, B7).

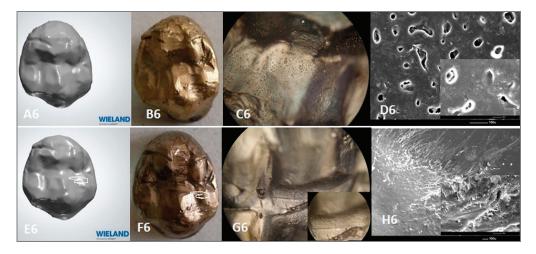


Fig. 8. Macro- and micro-surface analysis images for SC samples (Group 6) (left to right: CAD, DC, OM, SEM) after TC (A6-D6) and after ML (E6-H6). DC magnification at 2.5×, OM magnification at 4× and 10× and SEM magnification at 45× and 450×



Fig. 9. Macro- and micro-surface analysis images for G samples (Group 7) (left to right: CAD, DC, OM, SEM) after TC (A7-D7) and after ML (E7-H7). DC magnification at 2.5×, OM magnification at 4× and 10× and SEM magnification at 45× and 450×

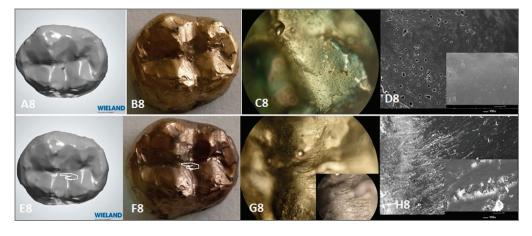


Fig. 10. Macro- and micro-surface analysis images for C samples (Group 8) (left to right: CAD, DC, OM, SEM) after TC (A8-D8) and after ML (E8-H8). DC magnification at 2.5×, OM magnification at 4× and 10× and SEM magnification at 45× and 450×

When the material was subjected to simulated-abrasion (ML), it maintained its occlusal contour and marginal integrity (Fig. 9E7, F7). The microscopic observations (OM and SEM) have revealed for TC aging method the presence of a rough surface with exposure and dislocation of some of the irregular particles (Fig. 9C7, D7). In the OM and SEM micrographs after ML method, one can see a roughen surface with distort occlusal morphology, parallel wear traces, micro-cracks and dislocation sites of the filler particles (Fig. 9 G7, H7).

Qualitative evaluation of the C resin inlays (Group 8) did not show any macro-structural modifications when the samples were subjected to thermal aging (Fig. 10A8, B8). After mechanical loading, the worn cuspal surface may be observed directly on the occlusal area (Fig. 10E8,F8). The micro-surface analysis revealed exposure of round-shaped particles, severe wear traces, micro-cracks within the material and debonding areas between fillers and resin matrix observed from 4× (OM method) up to 450× magnification (SEM method) (Fig. 10C8, D8, G8, H8).

4. Discussion

The tribological wear of dental biomaterials is a result of different factors, such as abrasion, erosion, fatigue or patient-related factors [4], [18], [25].

For a dental clinician, fast assessment of a fatigue-worn restoration status and decision to repair or replace it, is mainly based on the macro-surface evaluation devices present in the dental office (digital camera, intra-oral camera, etc.), and less on the micro-morphological details. Nevertheless, it is more recommended considering the results from both types of morphological analysis [4], [14].

One of the main factors which will clinically indicate a dental restoration to be replaced or repaired is the cyclic wear phenomenon [12]. The mechanisms to produce wear on dental structures and restorative materials may as well differ from one another or may be combined [4], [10], [12], [17], [25].

In this regard, our qualitative investigation was, firstly, based on the analysis of the surface details' features for both restoration methods (lab-fabricated resin composite inlays and direct restorative resin based composites) (Table 2), in order to better understand which of the extrinsic morphological elements may interfere and interact with the abrasion phenomenon. Secondly, intrinsic elements of restorative resin composites, such as type, shape and distribution of the

filler particles, composition of the resin matrix monomers and their intricate behaviour during the action of different oral agents (masticatory forces, moist environment, temperature changes, etc.) also played an important role, in our evaluation, to the overall performance outcome of a dental resin-based restoration (Table 2).

In this in vitro study, two-body wear behaviour of 8 dental restorative resin composites (four direct nanohybrid resin-based composites and four indirect resin composite inlays – two nanohybrid and two microhybrid, respectively) was tested using a dual-axis chewing simulator and a thermal cycler. The epoxy resin replicas of the restorations were evaluated for the macro- and micro-morphological surface features after in vitro testing procedures (TC and ML), with the following qualitative methods: 3D laser analysis (CAD system), DC, SEM and OM.

The macro-morphological analysis revealed that all the direct resin based biocomposites are more prone to violate the occlusal anatomical shape, marginal adaptation and surface polishability criteria (Table 2), compared with the lab-fabricated resin composite inlays. This may be explained by the differences in restoration methods (the resin composite Groups 1–4 were directly applied layer by layer in the MOD cavities; for the resin composite inlays (Groups 5–8), an impression was firstly used to pour the dies, the materials were incrementally applied in the cavity of the casts, polymerized using an oven and then, the inlays cemented in the MOD cavities with a luting agent), operator's skills during direct restorative procedures to redo the initial anatomical tooth morphology, use of different adhesive procedures for both types of restorations and different finishing and polishing systems used for direct MOD restorations and MOD inlays, respectively [10], [14], [21].

Regarding the micro-morphological criteria, our results showed a similar attrition behaviour between the P and VP samples, based on the presence of prepolymerised particles within their composition, which are able to protect the surface from excessive wear [10]. These results can be explained by a high percentage, type and shape of filler particles [16], [20] and also by the behaviour of the spherical particles and nano-clusters within the inorganic phase able to provide an even surface during abrasion [14], [23], [25]. Moreover, under the two-body wear conditions, the microphotographs' results of the VP restorations lead to similar type of scratches along the wear direction as shown in reference [10].

A resin-based composite material is a three-phase system, mainly composed of filler particles, resin matrix and the coupling agent used to bond the first two elements [7]. Each of these components is able to assure certain properties of the resin composite material. Attrition phenomenon between a natural tooth and a restorative material in combination with temperature changes, may alter these intrinsic abilities, thus, changing the properties of the whole structure.

During the macro- and micro-morphology assessment our findings showed a low degree of polishability and multiple surface structure defects for both K and BF samples after these had been subjected to the aging methods. One of the main reasons for the low surface properties found for K composite may be explained by the high content of water uptake of resin monomers (UDMA-DX-511 mixture) during aging procedures. Our findings (Fig. 5) are in agreement with reference [7]. When a "fluoride-releasing material" [4] is used to restore a stress-bearing cavity two queries are arisen: on the one hand, ability of fluoride release based on water uptake and recharge, and on the other hand, capacity to assure surface and intrinsic mechanical properties of the material in a given situation. According to the manufacturer, the BF material is considered a Giomer, because it has a high content of Surface Pre-Reacted Glass-ionomer (S-PRG) particles (0.01-4 µm) (able to release fluoride and recharge) [2]. It is known that fluoride-ion release mechanism is a very important element for restorations placed in the lateral teeth [4], although it was shown that, in time, it gives to hybrid ionomers a decreased resistance to wear and abrasion [4], [5]. Moreover, the material's organic phase is composed mainly of Bis-GMA and TEGDMA monomers [2], which makes this mixture prone to a significant water uptake [5], [7]. Thus, gathering the information about all the elements which compose the BF material and based on our findings during macro- and micromorphological evaluation, we came up with the idea that for this group a similar water sorption phenomenon with that from reference [5] might happened.

The degree of conversion is highly influenced by a higher filler particles fraction, type of resin matrix monomers and also by the type of polymerisation source [[4]]. Thus, using an external heat polymerisation source in addition to the light-curing, combined with a higher period of time of photo-polymerisation, gives the lab-fabricated resin composite materials the opportunity to offer to the future prosthetic restoration the ability to withstand higher occlusal attrition forces, temperature changes and to provide better surface details regarding anatomical shape, marginal adaptation and polishability compared with direct resin based composites.

Regarding the lab-fabricated inlays, PI material showed better wear behaviour compared with the above-mentioned direct resin composites and also with the other tested lab-fabricated resin materials. This surface behaviour may also be explained by the filler morphology (spherical and irregular particles) and the distribution of the particles within a range of 0.5–4 µm [17], [20]. Moreover, it is worthwhile to mention the presence of fine nano-particles arranged within nano-clusters (20–40 nm) [17], [20] which may also provide a higher filler compaction and thus, a better wear behaviour [7], [14].

A degree of surface flaws was also observed during macro- and micro-surface analysis for SC material (Fig. 8). It was thought that the resin-based multifunctional methacrylic acid esters matrix had absorbed a high amount of water during aging methods without any mechanical protection provided by the fillers. The reported percentage of filler particles (73 wt.%) of this indirect nanohybrid resin composite [19], and the filler morphology found (a combination of spherical and irregular particles with uniform distribution) [20] indicates a wear behaviour similar to microhybrid filled resin biocomposites.

Thermal cycling and especially, two-body wear revealed a similar fatigue wear behaviour for G and PI inlays (Figs. 7, 9) and better surface details for G material compared to SC and C resin composite inlays evaluated in this study. As was stated (Table 1), the G material is a micro-hybrid resin composite with 75 wt.% [6] and with irregular shape of filler particles (average size <2 µm) [20]. This type of wear behaviour is characteristic of these microhybrid filled resin composites due to the low distribution of the fillers and a large area between the particles, providing in this way a wider surface of the resin matrix to be exposed to abrasion and fatigue mechanisms [7], [10], [14], [20].

In a 3-year randomized clinical trial study, the SEM qualitative evaluation showed for the microfilled, micro- and nano-hybrid resin composite materials being tested a fatigue micro-crack behaviour and a capacity for pit formation during the clinical service, higher for micro-hybrid resin composite compared with nano-hybrid one. Furthermore, the same authors concluded that all the materials under study had developed similar cyclic fatigue patterns and cracks at "heavy OCAs" [14]. In our study, similar findings of cyclic fatigue wear patterns were observed between the nanohybrid SC material and the microhybrid C samples (Figs. 8, 10). The behaviour of C resin composite material may also be explained by the presence of round-shaped filler particles within its composition [3], [20]. It is also known that spherical particles are able to provide a lower wear rate of the natural antagonist tooth, but are not able to provide high mechanical strength and especially wear and cyclic fatigue strength compared with compact nano-hybrid particles [4], [7], [25].

5. Conclusion

The use of the two-body wear simulation method revealed important information about the behaviour of the dental resin-based composites when multiple oral factors are involved in lab-simulated conditions.

Within the limitations of this study careful conclusions may be drawn:

- the macro- and micro-morphological analysis showed different cyclic abrasion patterns among the test materials according to the filler percentage and distribution of the particles within the resin matrix. Furthermore, the 3D laser scan of the CAD system and the digital camera, illustrated macromorphological features which provided useful data for the clinical evaluation.
- regarding the direct resin-based composites, the P and VP materials showed a better behaviour to wear and cyclic fatigue mechanisms than the other two direct nanohybrid composites (K and BF).
- overall, the resin inlays showed a better adaptation and more finished and polished occlusal surfaces after in vitro aging procedures compared to the direct resin composite restorations. However, the SC nanohybrid resin inlay did not perform as expected compared to the rest of the test nanohybrid resin biocomposites, but had a similar surface behaviour with the other two microhybrid resin inlays (G and C) when they were subjected to simulated abrasion.

Acknowledgements

The authors want to express special thanks to Associated Professor MD PhD Codruta Nicola for the professional advices and support.

References

- [1] AMARAL F.L.B., COLUCCI V., PALMA-DIBB R.G., CORONA S.A.M., Assessment of in vitro methods used to promote adhesive interface degradation: A critical review, J. Esthet. Restor. Dent., 2007, Vol. 19, 340–354.
- [2] Beautifil II Product brochure. http://www.shofu.com/shofu_images/Literature/beautifil%20ii%20brochure.pdf. Accessed 2 May 2013.

- [3] Ceramage Product brochure. http://www.shofu.com/ shofu_images/Literature/ceramage%20brochure%201.08%20 v2.pdf . Accessed 2 May 2013.
- [4] CURTIS R.V., WATSON T.F., Mixed-methods approach to wear evaluation in posterior composite dental restorations, [in:] Dental biomaterials. Imaging, testing and modelling, Woodhead Publising Limited and CRC Press LLC, Cambridge 2008.
- [5] GONULOL N., OZER S., TUNC E.S., Water sorption, solubility, and color stability of giomer restoratives, J. Esthet. Restor. Dent., 2014, Vol. 21,1–6. DOI: 10.1111/jerd.12119. [Epub ahead of print].
- [6] Gradia-Product brochure. http://www.gceurope.com/pid/67/manual/en Manual.pdf. Accessed 2 May 2013.
- [7] ILIE N., HICKEL R., Resin composite restorative materials, Aust. Dent. J., 2011, Vol. 56, 59–66.
- [8] ISO. Dental materials guidance on testing of wear. Technical Specification, 2001, No. 14569-2, Part 2.
- [9] Kalore-Product brochure. http://www.kalore.net/GCA_ KALORE Technical Manual.pdf. Accessed 2 May 2013.
- [10] KOOTTATHAPE N., TAKAHASHI H., IWASAKI N., KANEHIRA M., FINGER W.J., Morphological features of composite resin surfaces after two- and three-body wear simulation, World J. Dent., 2012, Vol. 3, 221–228.
- [11] LAMBRECHTS P., DEBELS E., VAN LANDUYT K., PEUMANS M., VAN MEERBEEK B., *How to simulate wear? Overview of existing methods*, Dent. Mater., 2006, Vol. 22, 693–701.
- [12] LEE A., HE L.H., LYONS K., SWAIN M.V., Tooth wear and wear investigations in dentistry, J. Rehab., 2012, Vol. 39, 217–225.
- [13] MILEWSKI G., Numerical and experimental analysis of effort of human tooth hard tissues in terms of proper occlusal loadings, Acta Bioeng. Biomech., 2005, Vol. 7(1), 47–59
- [14] PALANIAPPAN S., BHARADWAJ D., MATTAR D.L., PEUMANS M., VAN MEERBEEK B., LAMBRECHTS P., Three-year randomized clinical trial to evaluate the clinical performance and wear of a nanocomposite versus a hybrid composite, Dent. Mater., 2009, Vol. 25, 1302–1314.
- [15] POPOVICI A., FODOR O., MOLDOVAN M., ROMAN A., BORZEA D., "In vitro" study of a new composite biomaterial for indirect restorations, Optoelectron. Adv. Mater. Rapid Comm., 2008, Vol. 2, 891–894.
- [16] Premise Product brochure. http://www.kerrdental.com/ kerrdental-composites-premise-techinfo-2. Accessed 2 May 2013.
- [17] Premise Indirect Systems, Kerr Laboratory. http:// www.kerrlab.com/ dfu. Accessed 2 May 2013.
- [18] SAJEWICZ E., KULESZA Z., A new tribometer for friction and wear studies of dental materials and hard tooth tissues, Tribol. Int., 2007, Vol. 40, 885–895.
- [19] Signum Ceramis Product brochure. http://webmedia. kulzer-dental.com/media/hkg/downloads_new/signum_2/signum_ceramis/GBA_Signum_ceramis_INT.pdf. Accessed 2 May 2013.
- [20] SOANCA A., ROMAN A., MOLDOVAN M., PERHAITA I., TUDORAN L.B., ROMÎNU M., Study on thermal behaviour, structure and filler morphology of some indirect composite resins, Dig. J. Nanomater. Bios., 2012, Vol. 7, 1071–1081.
- [21] SOANCA A., ROMINU M., MOLDOVAN M., BONDOR C.I., NICOLA C., ROMAN A., Microscopic evaluation of the

- interface between composite biomaterials and dentin biostructure, Dig. J. Nanomater. Bios., 2011, Vol. 6, 349 –358.
- [22] STEINER M., MITSIAS M.E., LUDWIG K., KERN M., *In vitro* evaluation of a mechanical testing chewing simulator, Dent. Mater., 2009, Vol. 25, 494–499.
- [23] TURSSI C.P., DE MORAES PURQUERIO B., SERRA M.C., Wear of dental resin composites: Insights into underlying proc-
- esses and assessment methods A review, J. Biomed. Mater. Res., 2003, Vol. 65, 280–285.
- [24] Venus Pearl Product brochure. http://webmedia.kulzer-dental.com/media/hkg/downloads_new/venus_5/venus_pearl_1/ GBA_Venus_Pearl_INT.pdf. Accessed 2 May 2013.
- [25] WOJDA S., SZOKA B., SAJEWICZ E., *Tribological characteristics of enamel–dental material contacts investigated in vitro*, Acta Bioeng. Biomech., 2015, Vol. 17, 21–29.