

IN VITRO EVALUATION OF THE INFLUENCE OF CONTRACTION STRENGTH IN POLYMERIZATION OF RESTAURATIVE COMPOSITES ON ADHESIVE CAPACITY OF ADAPTION SYSTEMS

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ABSTRACT

Modern Restorative Dentistry today successfully capitalizes on the advances in the rapid evolution of adhesive systems, both in terms of materials and application techniques. Current patient requirements for aesthetics of restorations, as well as the development of adhesive techniques, have imposed composite diacrilic resins (DRCs) as selective materials for direct composite restorations. The contraction of polymerization as well as the stress of the polymerization contraction manifested inside the composite can compromise adhesion between the obturation and the dental tissue, with the appearance of the marginal microinfiltration phenomenon with all the known negative effects such as the occurrence of marginal stains, secondary caries and even pulp necrosis. Numerous studies on the adhesion of materials to dental structures have the objective of knowing and eliminating as far as possible the causes of degradation of the adhesive interface. In this note are also written the authors of this article, whose experimental results provide useful information regarding the influence and evaluation of the contraction stress on the polymerization of the restorative composites and different adhesive system.

Keywords: adhesion, stress of contraction, polymerization, composite, adhesive system

INTRODUCTION

Composite materials commonly used today have a composition based on dimethacrylic monomers which have a major disadvantage regarding the contraction they produce during the polymerization. The resin system contracts during polymerization due to the formation of a macromolecular network of various distinct monomers, involving the conversion of intermolecular distances of approximately 0.3-0.4 nm into covalent bonds of approximately 0.15 nm length [1].

Generally, the polymerization contraction values of the composites are in the 2.6% -7.1% range [2], the current composites having a contraction of 2% -3% and the ADA

(American Dental Association) specification requires a maximum of 3% [3 , 4, 5]. The stress of the polymerization contraction is a factor responsible for the degradation of the adhesive interface and ultimately for the reduction of the longevity of the composite restorations [6, 7], but most of the studies about marginal microinfiltration associated with the direct composite restorations attribute the infiltration to the stress of the polymerization contraction, but there is the possibility that its negative effect on marginal adaptation may be overestimated and incorrectly correlated with the values of the cusps deformations [8, 9, 10, 11].

THE AIM OF THE STUDY

In the present study, two experimental composites were investigated: a composite having as a basic monomer a urethane derivate of bis-GMA1, and the second conventional Bis-GMA monomer. The purpose of the study is to perform determinations for the evaluation of the polymerization contraction stress at the tooth-fill interface. The fillings using the two experimental composites based on different base monomers were performed on the upper premolars with MOD cavities, the evaluation of the stress of the polymerization contraction at the tooth-restoration interface was made by applying two tests:

1. Evaluation of the marginal sealing on the enamel threshold by testing marginal microinfiltration with an organic dye (basic

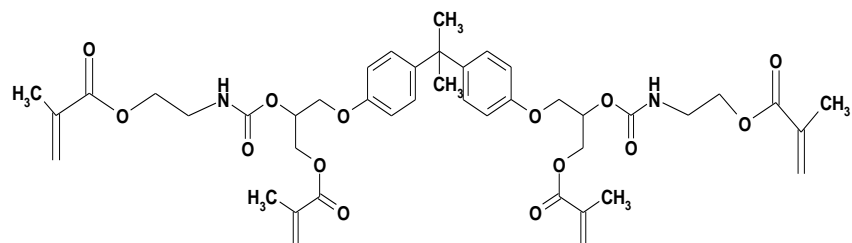
fluxine) and optical microscope analysis

2. Evaluation of the integrity of the adhesive interface on enamel and dentin by Fe-SEM analysis.

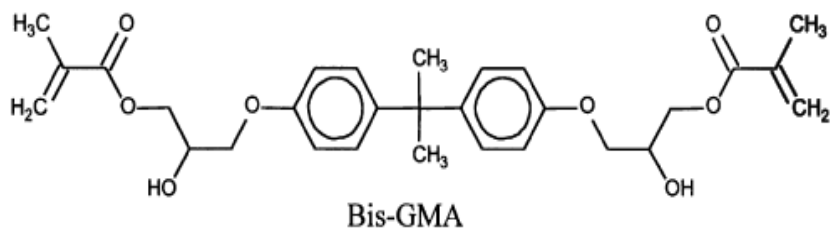
MATERIALS AND METHODS

A bis-GMA urethane derivate monomer, named bis-GMA1, prepared by the "Petru Poni" Macromolecular Chemistry Institute from Iași, was used by the addition reaction between the hydroxyl group of the bis-GMA monomer and the methacryloyloxyethylisocyanate [12].

The classical Bis-GMA monomer was purchased from Sigma Aldrich Chemical Co. and was used as a control. The molecular structure of the base monomers used is shown in Figure 1



Bis-GMA1



Bis-GMA

Fig. 1. Molecular structure of Bis-GMA1 and Bis-GMA monomer[5]

Two Diacrylic Resins were prepared based on the investigated monomers combined with TEGDMA in a 65/35 dilution ratio (Bis-GMA1 / TEGDMA and Bis-GMA / TEGDMA) with 80% charging of hybrid inorganic particles (glass of Barium oxide, Quartz and Colloidal silica). The preparation of the resins was carried out in the Department of Polymer Composites belonging to the "RalucaRipan" Chemistry Research Institute in Cluj-Napoca, respecting the direct composites manufacturing protocols and the requirements of the international standards for composite materials for dental use ISO 4049: 2000. 20 supernumerary premolars extracted for orthodontic purposes were selected for the study, which were visualized without caries, hypoplastic defects or fisures. The teeth were

cleaned, the soft deposits and tartar removed, and kept until preparation in 0.9% NaCl solution containing 0.02% azide sodium at 4 ° C. The maximum bucco-palatal size (BPS) of each tooth was measured, and the values obtained were used to distribute the teeth in two groups of 10 teeth, so that the mean DVP between groups varied by no more than 5% (Table 2). Each tooth was fixed in an autopolymerizable acrylic resin (Duracryl Plus: SpofaDental, CZ) in a 15 mm stainless steel cube with a 12 mm diameter internal cylindrical structure. The thus fixed teeth were kept in double distilled purified water at 23 ± 1 ° C except for the times when the research protocol required isolation conditions against moisture.

Table 2: Mean size of premolars (µm) taken into study (statistically significant difference p<0.05)

Group 1 (n=10)		Group 2 (n=10)		p (Kruskall Wallis)
Mean	S.D.	Mean	S.D.	
9.47	0.53	9.36	0.64	0.87

Standardized MOD cavities [13,15] were prepared with a red ring diamond bur (FG837F014, Meisinger, Germany) attached to a NSK PANA AIR turbine (NSK, Tochigi, Japan) at a speed of 380,000 rpm with continuous water cooling, as shown in Figure 2. The bucco-palatal dimension of the proximal cavities was prepared at 2/3 of the BPS of the tooth, with the gingival threshold

of 2 mm parapulpal depth and 1 mm above the CEJ. The occlusal isthmus was prepared at half the size of the BPS, at a standard depth of 3.5 mm from the tip of the palatal cusp. The edge of the cavity was prepared at 90 ° and all internal angles were rounded and the axial walls parallel to each other. Both premolar groups were restored layered with the composite materials taken into study in combination with an adhesive system photopolymerized using the Demi LED lamp (Kerr) with an oscillating light intensity

between 1100 mWcm⁻² and 1330 mWcm⁻². The restoration assumed their layered application, a first layer being approximately 1 mm horizontally applied on the MOD cavity so as to obtain a good adaptation of the of the composite to the dentinal walls of the cavity; the following 8 layers of composite, 3 for the each vertical cavities and 2 for the occlusal cavity were applied in a triangular shape of

about 2 mm thickness without touching both opposing walls of the proximal cavity. The MOD cavity was restored in the following order: the M cavity, the D cavity, then the O cavity. Each layer was photopolymerized 20 s with the DEMI lamp from a distance of 2 mm above each cusp.

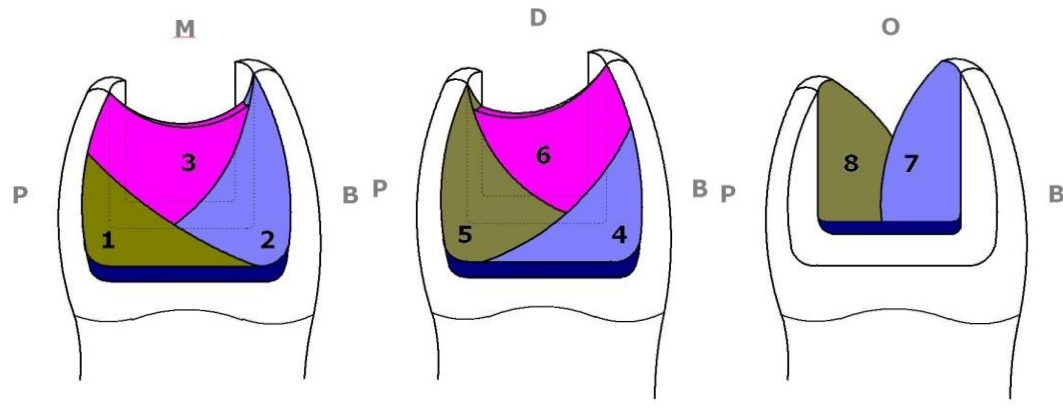


Fig. 2. The layered technique used in the current study

Legend. M: mesial cavity, D: distal cavity, O: occlusal cavity, B: buccal cusp, P: palatal cusp, 1-8: oblique layers of composite, dark blue horizontal layer: horizontal layer of composite

In the in vitro experiment, the effects of the contraction stress on the polymerization of the studied materials were followed on natural teeth to simulate the clinical reality as accurately as possible during their photopolymerisation. The adhesion of the two experimental composites was achieved with an adhesive system with

etch- and- rinse, in three steps, OptiBond FL (Kerr), chosen from a previous microinfiltration study of restorations with nanoparticle composite Premise Packable (Kerr) with two different adhesive systems from the same Kerr company, but with different adhesion strategies (OptiBond FL,

OptiBond All-In-One).

The evaluation of marginal microinfiltration was carried out at the Department of Dentures and Prosthetics Technology, UMF "Carol Davila", Bucharest. The teeth were cut mesio-distally with a water-cooled microtome (Isomet Low Speed Saw, Buehler Ltd) to obtain a 1.5 mm thick section at the mesio-distal groove of the tooth. Each section was evaluated at the Olympus CKX31 (Olympus America Inc.) at a 40x magnification to measure marginal microinfiltration (µm) with a QuickPhoto Micro 2.2 software (Olympus Inc.). For each restoration, the entire MOD cavity interface was analyzed with the overlying restoration in

order to identify the marginal microinfiltration of the basic fuxine. After the analysis of the sections by optical microscopy, three sections of each group were randomly selected, which were examined by electron microscopy Fe-SEM type. The integrity of the tooth-restoration adhesive interface was analyzed using the FEI - Inspect S 50 (FEI Co., Oregon, USA) electronic microscope from the National Research and Development Institute for Lasers, Plasma and Radiation Physics (INFLPR), Magurele, Romania.

EXPERIMENTAL RESULTS AND DISCUSSIONS

On the evaluated sections at the optical microscope, it was observed that the basic Fuxin marginal microinfiltration on the enamel threshold was absent in all restorations, regardless of the type of the basic monomer of the investigated composites. (Fig. 3, 4). On all the analyzed sections it was observed both the integrity of the adhesive interface between the enamel and the adhesive, as well as between the adhesive layer and the overlying composite.

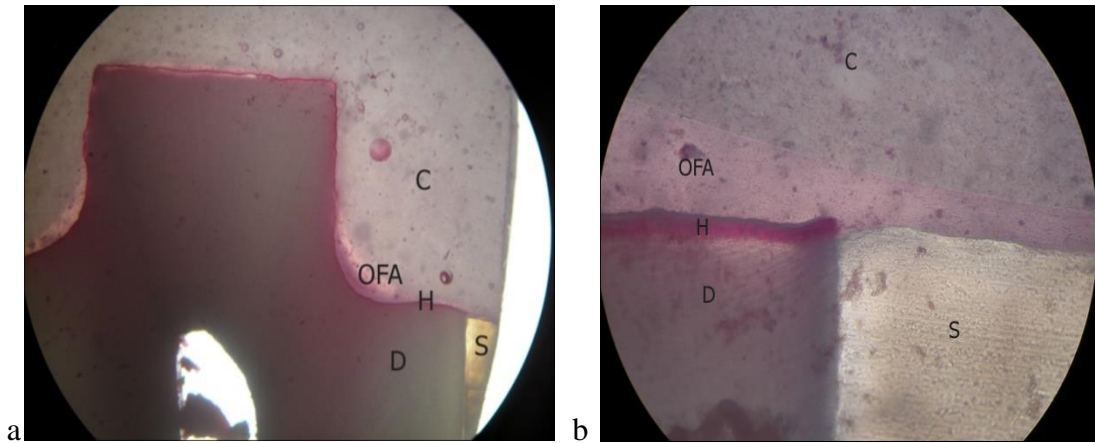


Fig. 3. Optical microscopy images (a-40x magnitude, b-100x magnitude) revealing the absence of microinfiltration of the dye on enamel at Bis-GMA1-based restorations; S-enamel, D-dentin, C-composite, OFA-OptiBond FL Adhesive, H-hybrid layer.

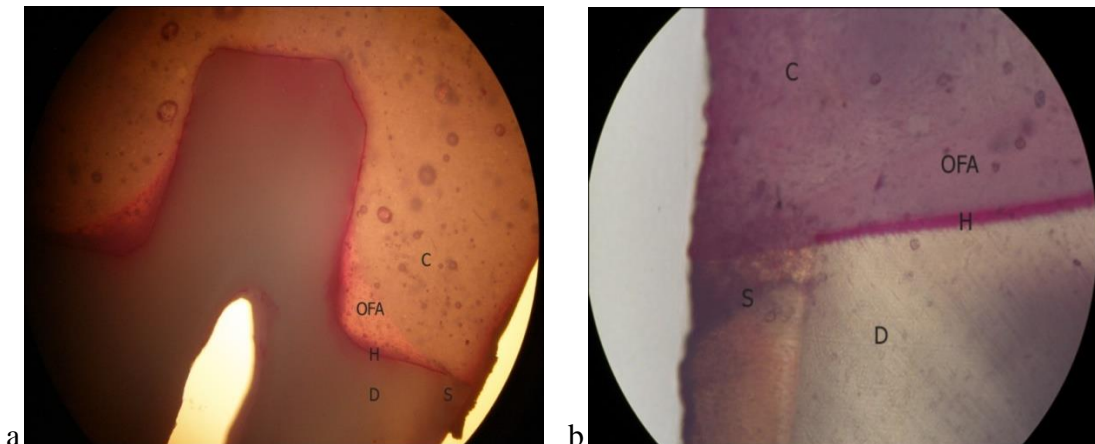


Fig. 4. Optical microscopy images (a-40x magnitude, b-100x magnitude) revealing the absence of microinfiltration of the dye on enamel at Bis-GMA-based restorations; S-enamel, D-dentin, C-composite, OFA-OptiBond FL Adhesive, H-hybrid layer.

The 50X magnification Fe-SEM electron microscopy images illustrate the interface between the composite and the dental structures, but also the integrity of the restoration and the underlying tooth. In the images made for the Bis-GMA1 composite, in some areas there is a separation between the restoration and the dentine, between the restoration and the enamel being observed the integrity of the adhesive interface. In the images corresponding to a bis-GMA filling, the continuous passage between the restoration and both types of dental hard tissue

of the prepared cavity and the integrity of the restoration are observed (Figure 5). Fe-SEM electron microscopy images at 1000X magnification illustrate the adhesive interface between dental hard tissues and the obturation, highlighting the interface between the enamel / dentine and the corresponding OptiBondFL Adhesive filling layer, but also between them and the overlying composites.

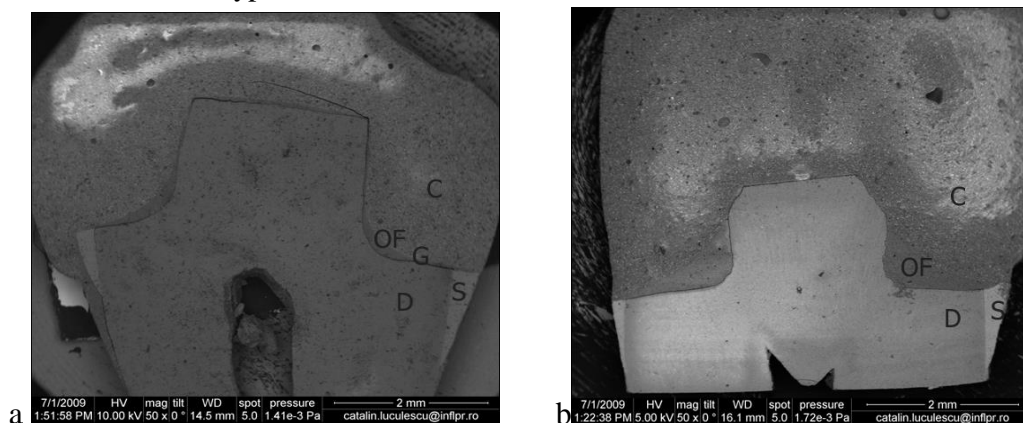


Fig. 5. Fe-SEM electron microscopy micrographs at 50X magnification of restorations based on Bis-GMA1 (a) and Bis-GMA (b). S-enamel, D-dentin, C-composite, OF-OptiBond FL, G-gap remaining enamel on the cervical threshold are observed.

For both types of composite, the integrity of the adhesive interface between the enamel adhesive layer (where is observed only OptiBond FL Adhesive in this magnification) - the overlying composite is confirmed. At the selected image corresponding to a Bis-GMA1 filling, the enamel adhesion is integral, but fissures in the

remaining enamel on the cervical threshold are observed. The crack extends between the enamel-dentine junction and the point on the outer enamel wall of the preparation, where the acid has reached, and the adhesion of the overhang area is achieved. (Fig.6).

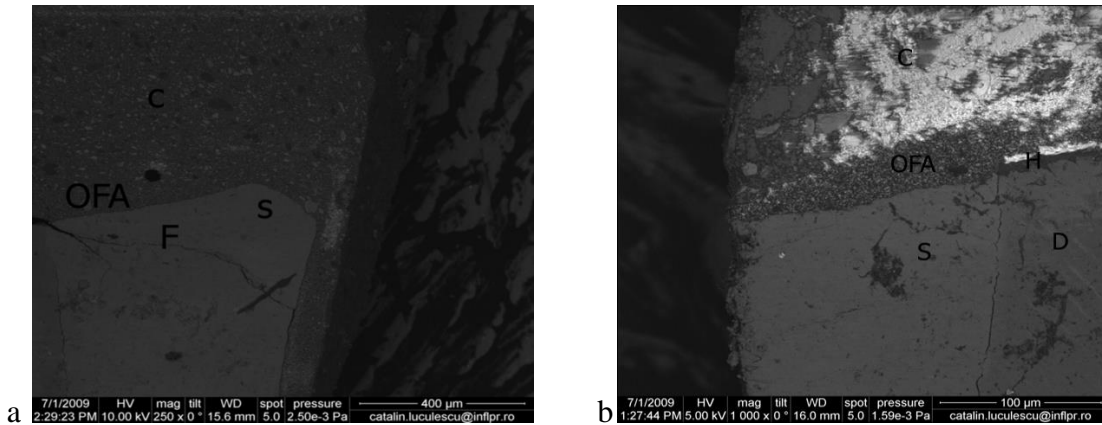


Fig.6.Fe-SEM microscopy micrographs at 1000X illustrating the enamel-composite adhesive interfaces of Bis-GMA1 (a) and Bis-GMA (b) restorations.S-enamel, D-dentin, C-composite, OF-OptiBond FL, G-gap.

On Fe-SEM 1000X micrographs illustrating the integrity of adhesion between dentin and composite (Fig.7), it is noted that the OptiBond FL adhesive system ensures the continuous passage between dentine and composite only for the Bis-GMA based

composite. On the images corresponding to the dentine adhesion of Bis-GMA 1 fillings, cohesive tearing of the adhesive layer is observed by compromising the hybrid layer-hydrophobic resin junction.

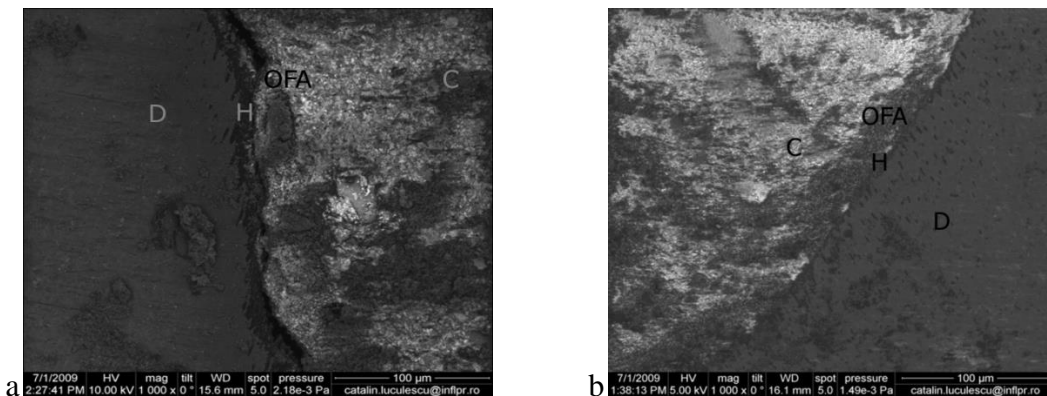


Fig.7.Micrographs of Fe-SEM microscopy at 1000X magnification illustrating the dentin-adhesive-composite layers of the Bis-GMA1 (a) and Bis-GMA (b) based restorations.S-enamel, D-dentin, C-composite, OF-OptiBond FL, G-gap.

The results of both electronic microscopy and optic microscopy confirmed the perfect adhesion of the restorative composites on enamel, irrespective of their base monomer, through the OptiBond FL adhesive system. It is known that if the stress of the polymerization contraction is greater than the adhesion force between the filling and the tooth, then the adhesive layer breaks [6]. The adhesion mechanism, themorpho-structural particularities and the more hydrophobic character of the enamel, gave this enamel adhesive system a traction resistance superior to the traction force corresponding to the stress of the polymerization contraction of the two studied composites. The polymerisation contraction or its rate of production on the Bis-GMA1 based composite resulted in a higher polymerization contraction stress than that produced by Bis-GMA, such that in the dentine, this bis-GMA based composite affected the cohesion between the superficial boundary of the hybrid layer and the OptiBond FL Adhesive adhesive resin.

This interface between the hybrid layer and the overlying sealing resin was the area of minimum resistance to the dentine, as the enamel cracks constituted evidence of minimal resistance in the 1 mm enamel left over the cemento-enamel junction. The initial form under which OptiBond FL was marketed, OptiBond, had a photopolymerization protocol between the primer and the adhesive resin, in the new product prospectuses the manufacturer recommended not to light cure between the two layers. Probably the breakdown of cohesion in the adhesive layer was primarily due to the excessive polymerization contraction of the bis-GMA1-based composite, the incompatibility of the hydrophilicity between the two components of the OptiBond FL adhesive system, having to be verified in further studies. The stress of

polymerization contraction generated by the bis-GMA-based composite was not greater than the adhesion force of the adhesive system used on both enamel and dentin.

The results of this study recommend the OptiBond FL system for direct composite restorations, with the ability to withstand the stress of the polymerization contraction, while retaining the marginal integrity of the fillings.

CONCLUSIONS

The researches aimed at assessing the stress of the polymerization contraction generated by two experimental composites at the adhesive interface between the restoration and the dental hard tissue by testing the marginal microinfiltration. The results obtained allow the following conclusions to be made based on observations and measurements:

1. Using the self-etch adhesive system in one step, the OptiBond All-In-One system did not perform significantly better than the three step etch-and-rinse system OptiBond FL, but there was a significant reduction in marginal microinfiltration by using the self-etch adhesive system, on both enamel and cement, in one step.
2. The adhesive capacity of the OptiBond All-In-One system to marginal seal the composite restorations proved to be more efficient at the cement threshold than at the enamel threshold.
3. The adhesive capacity of OptiBond FL system, etch-and-rinse in 3 steps, demonstrates that this is the most effective adhesive system for the marginal closure of composite restoration with the cervical threshold on enamel
4. The use of urethane-derived composite material (Bis-GMA1) has only

benefited from the reduction of the cusp extension without increasing or reducing the cuspidian flexion (the stress of the polymerization contraction in the dental walls) compared to a conventional composite based on Bis-GMA.

5. The stress of the polymerization contraction generated by the Bis-GMA1-based composite at the adhesive interface did not affect the tightness of the marginal seal on the enamel but caused the cohesive breakage between the superficial boundary of the hybrid layer and the overlying sealant resin in the dentine.

The results of our study demonstrate that only conventional Bis-GMA-based composite generates an acceptable polymerization contraction stress in the whole composite-adhesive layer-dental structures complex; The stress of polymerization contraction at the adhesive interface level generated by the conventional Bis-GMA composite was not greater than the adhesion force between the interphases of the dental tissue-hybrid layer-sealing resin-composite.

References

1. Peutzfeld A., Resin composites in dentistry: the monomer systems. *Eur J Oral Sci.* 1997;105:97-116
2. Feilzer AJ, De Gee AJ, Davidson CL – Curing contraction of composites and glass-ionomer cements. *J Prosthet Dent.* 1993;9:2-5
3. Choi KK, Ferracane JL, Hilton TJ, et al – Properties of packable dental composites. *J Esthet Dent.* 2000;12:224-234
4. Romînu M, Bratu D, Lakatos S, et al – Polimerizarea în stomatologie. Timișoara: Ed. Brumar; 2000
5. Sakaguchi RL, Peters MCRB, Nelson SR, et al – Effects of polymerization contraction in composite restorations. *J Dent.* 1992;20(3):178-182
6. Davidson CL, Feilzer AJ – Review. Polymerization shrinkage and polymerization shrinkage stress in polymer-based restoratives. *J Dent.* 1997; 25:435-440
7. Bausch JR, De Lange K, Davidson CL – Clinical significance of polymerization shrinkage of composite resins. *J Prosthet Dent.* 1982;48:59-67
8. Abbas G, Fleming GJP, Harrington E, et al – Cuspal movement in premolar teeth restored with a packable composite cured in bulk or incrementally. *J Dent.* 2003;31:437-444
9. Cara RR, Fleming GJP, Palin WM, et al – Cuspal deflection and microleakage in premolar teeth restored with resin-based composites with and without an intermediary flowable layer. *J Dent.* 2007; 35(6):482-489
10. Fleming GJP, Cara RR, Palin WM, et al – Cuspal movement and microleakage in premolar teeth restored with posterior filling materials cured using ‘soft-start’ polymerisation. *Dent Mater.* 2007;23:637-643
11. Palin WM, Fleming GJP, Nathwani H, et al – In vitro cuspal deflection and microleakage of maxillary premolars restored with novel low-shrink dental composites. *Dent Mater.* 2005;21:324-335
12. Buruiana T, Buruiana EC, Melinte V, et al – New urethane dimethacrylates for testing in dental applications. *J of Optoelectronics and Adv Mat* 2008; 10(4):969-974
13. Versluis A, Tantbirojn D, Pintado MR, et al ., Residual shrinkage stress distributions in molars after composite restoration, *Dent Mater.* 2004; 20:554–564
14. Van Meerbeek B, Peumans M, Poitevin A, et al – Relationship between bond-strength tests and clinical outcomes. *Dent Mater.* 2010;26(2):100-121
15. Vîrlan C, Dimitriu B, Stanciu D, et al – Situația actuală a adeziunii la structurile dure dentare, Ghid de adeziune amelo-dentinară pentru restaurări estetice dentare: 2011, (www.SSER.ro)

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