INFLUENCE OF STORAGE TIME IN WATER ON THE INTEGRITY OF ADHESIVE INTERFACE IN RESIN COMPOSITE RESTORATIONS

ABSTRACT

When immersed in oral fluids, water absorption by the restorative resin composite material can occur, which is identified by some researchers as one of the causes of loss of aesthetic features and reduction of mechanical properties over time. On the other hand, some authors have suggested that the fluids sorption may contribute to the reduction of shrinkage stress generated at the adhesive interface and reduce the width of gaps. The aim of this study was verifying if the storage time in water of restorations carried out with different filling techniques could influence on the integrity of tooth-restoration adhesive interface. Eighteen cavities were built in bovine incisors and they were divided into 3 groups after the adhesive procedure: group B ("Bulk") received one single increment of light-cured resin composite; group I ("Increments") received the same composite in three oblique increments; and the group B+S ("Bulk + Self-cured resin composite"). The last one firstly received a flowable, self-cured resin composite, and then, it was inserted one single increment of light-cured composite. After 48 h of storage, the restorations were sliced, the first measurement was accomplished, and the analysis of the adhesive interface was made each 30 days over nine months of immersion in water. The results were subjected to a split-plot analysis of variance and Tukey's test. It was not verified significant influence of immersion time in water on the gap width, or regular increase or decrease of percentage interface free of gaps over time for any of three filling techniques. Some hypothesis could explain this occurrence, such as gain of mass without significant increase in the volume; the expansion of restoration in directions that did not contributed to the gaps closure; and the simultaneous occurrence of hygroscopic expansion and hydrolytic degradation of the resin processes.

XAVIER, Tathy Aparecida*
MEDEIROS, Igor Studart*
ROSA, Ecinele Francisca*
BALLESTER, Rafael Yague*

KEYWORDS

Composite resins. Dental restoration failure. Permanent dental restorations. Dental materials.

Department of Dental Materials, School of Dentistry, University of Sao Paulo, Sao Paulo, SP, Brazil*
Correspondence: tathy@usp.br (XAVIER TA)

| **Received** 14 May 2013 **Received in revised form** 29 May 2013 **Accepted** 09 Jun 2013

INTRODUCTION

The increase in the use of resin composite as a restorative material in Dentistry is mainly due to its aesthetic features and the possibility of making more conservative cavity preparations. Many studies have found failures of resin composite restorations due to the loss of integrity of the adhesive interface. These failures can lead to events such as microleakage and, consequently, post-operatory sensitivity, marginal discoloration and secondary caries.

The polymerization shrinkage has been studied by many research centers as one of the main causes of the loss of adhesion between the restoration and the tooth walls. As the restorative material shrinks adhered to the more rigid remaining tooth, stresses are developed in the tooth-restoration complex. If such stresses reach values higher than the adhesive strength at the interface, the restoration can detach from tooth and gaps can be created.

Although dental resin composites are made of inert ceramic filler and an organic matrix consisting of large, hydrophobic molecules, the sorption of fluids can occur when they are in the oral cavity. This event is identified by some researchers[1-3] as one of the causes of resin composite restoration failures related to loss of aesthetic features and reduction of mechanical properties. On the other hand, some findings suggested that the fluids sorption may lead to a plasticizing of the organic matrix and/or to its hygroscopic expansion, which can reduce the shrinkage stresses generated at the adhesive interface[4]. Furthermore, it was reported that the interfacial gaps can be closed or reduced by the hydroscopic expansion[1].

In view of these findings, the aim of this study was verifying if the storage time in water of restorations carried out with three different filling techniques could influence on the integrity of tooth-restoration adhesive interface.

MATERIAL AND METHODS

Eighteen cavities were built with dimensions: 3.5 x 2.5 x 1.5 mm, in the cervical region of proximal faces of bovine incisors, with the cervical margin in root dentin. All the cavities were etched with phosphoric acid (37%) for 15 s, followed by washing in running water for 20 s and drying with absorbent paper. After the acid etching, the multi-bottle adhesive system Adper Scotchbond Multi-Purpose (3M ESPE, St Paul, MN, USA) was applied in all the cavities. The steps were carried out as follows: (1) primer application for 45 s under agitation; (2) application of air jet to evaporate the solvent; repetition of steps (1) and (2) until obtain a shiny surface with no more liquid movement; (3) adhesive application and uniformization of adhesive thickness with air jet; (4) photo-activation for

20 s (600 mW/cm^2).

The cavities were divided into 3 groups, according to the technique of composite insertion. The cavities of group B ("Bulk") received the light-cured resin composite Tetric Ceram (Ivoclar Vivadent AG, Schaan, Liechtenstein) in a single increment. The Group I ("Increments") received the same resin composite in three oblique increments. The group B+S ("Bulk + Self-cured resin composite") firstly received the flowable, self-cured resin composite Bisfil 2B (Bisco Dental Products Canada Inc., Richmond, BC Canada), which covered the cavity walls, and then it was inserted a single increment of the same light-cured resin composite used on the other groups. All the increments of Tetric Ceram resin composite were photo-activated for 60 s (600 mW/cm^2).

After 48 h of storage in distilled water at 37 ^{o}C, the restorations were cut from the incisal towards to the cervical margin using a cutting machine (Isomet 1000, Buehler Ltd., Lake Bluff, IL, EUA) equipped with a diamond disc (0.3 mm of thickness) water- lubricated.

One of the two slices obtained from each restoration was embedded in self-cured acrylic resin (refrigerated by immersion in water) to have the tooth-restoration interface analyzed. The cutting surface was burnished with sandpaper (granulation from 600 to 2000) and etched with phosphoric acid (37%) for 15 s to eliminate the smear layer generated during the process. In order to eliminate collagen and microorganisms in the interface and to facilitate the visualization of gaps, the slices were immersed in a sodium hypochlorite solution (2.5%) for 10 min in an ultrasound vat, followed by 10 min of immersion in distilled water, also in the ultrasound vat.

In order to observe the gaps, it was not used a scanner electron microscope because it would require vacuum, causing loss of water from dentin and composite resin, which could alter the adhesive interface integrity. Then, it was used an optical microscope of a microhardness tester (Shimadzu HMV2, Shimadzu Scientific Instruments, Inc., Columbia, Maryland, USA) to analyze the interface under a magnification of 400x, with a method already published in the literature[5, 6].

Considering that, after the detachment, the gap width could depend more on the amount of polymerization shrinkage of the specific composite than on the stresses generated at the interface during the contraction, besides the gap width mean, it was measured the percentage of interface free of gaps for each filling technique.

The measurements of gap width mean and percentage of interface free of gaps were accomplished after the initial 48 h and in each 30 days over nine months of storage in distilled water at 37 ^{o}C. Before each analysis, the slices were subjected to cleaning with a sodium hypochlorite solution (2.5%) for

10 min in an ultrasound vat, followed by 10 min of immersion in distilled water, also in the ultrasound vat in order to eliminate impurities accumulated in the adhesive interface.

The results were subjected to a split-plot analysis of variance, since the data obtained over time for each slice were dependent, and Tukey's test, with an adopted global level of significance equal to 5%.

RESULTS

With respect to the gap width mean, it was not detected any significant difference for technique and period, or for the interactions technique x period, region x period and region x period x technique (table 1). There was not specific filling technique in which the gap width mean was significantly influenced by storage time in water. It was noticed a significant difference among the results obtained according to the cavity region and interaction technique x region. The Tukey's test identified significant differences among all the regions of the cavity for the techniques I and B+S, with the largest gap for the pulpal wall, and the smallest width at cervical wall (the one with enamel and dentin). The Tukey's test also showed that the significant interaction technique x region was due to the behavior of group B, that was the only one in which the gap width mean was similar in the regions incisal and pulpal (figure 1).

Regarding to the percentage of interface free of gaps, the analysis of variance detected a significant difference among the periods of immersion (table 2). However, the Tukey's test detected a significant difference only for the fifth month of storage, without a reduction or increase in a regular sequence over the months. According to the Tukey's test, the last month showed a percentage of interface integrity similar to the one measured in the first month for all the filling techniques.

DISCUSSION

Some authors stated that gaps in tooth-restoration interface may be gradually closed by the hygroscopic expansion of restorative material[1], and the water sorption is one of the mechanisms of relief of polymerization stress[4, 7, 8]. Some studies have found that the water sorption causes an expansion of the restorative material until an equilibrium value[7, 9], but the process is slow because the diffusion of water within the resin matrix decreases with the increase of water concentration inside the composite. A study[10] found that most of the hygroscopic expansion occurs in the first two weeks, with the balance achieved in about eight weeks. Apart from delayed repair of some failures, there are some adverse effects related to the water sorption, such as reduction of mechanical properties and staining[1, 11], though some authors[12] have found

Table 1. Split-plot analysis of variance of the gap width mean according to the filling technique (B, I or B+S), period of immersion (months) and cavity region (incisal, pulpal and cervical).

Source of variation	DF	SS	MS	(F)	p value
Technique (tech)	2	34.51	17.25	0.14	0.87
Residue I	15	1829.58	121.97		
Main block	17	1864.09	139.23		
Period (per)	8	91.88	11.48	1.04	0.41
Interaction tech x per	16	121.04	7.56	0.69	0.80
Residue II	120	1325.22	11.04		
Sub-block I	144	1538.14	30.09		
Region (reg)	2	2738.84	1369.42	85.68	**0.00(*)**
Interaction reg x tech	4	306.35	76.59	4.79	**0.00(*)**
Interaction reg x per	16	296.03	18.50	1.16	0.30
Interaction reg x per x tech	32	88.13	2.75	0.17	0.99
Residue III	270	4315.33	15.98		
Sub-block II	324	7744.68	1483.25		
Total	485	11146.91	1652.57		

* Means significant difference at the level of 5%.

Figure 1. Gap width mean (µm) according to the cavity region for the three filling techniques.

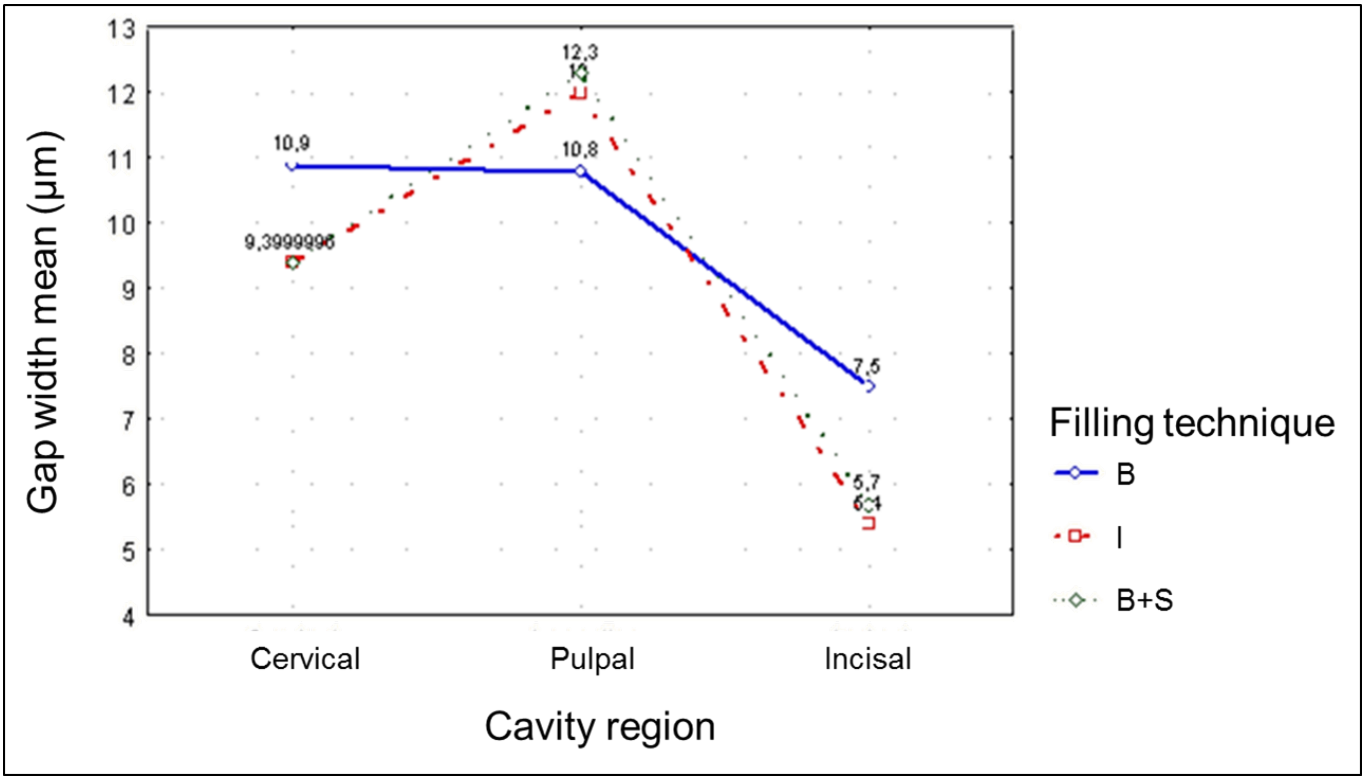

Table 2. Split-plot analysis of variance of the percentage of interface free of gaps according to the filling technique (B, I or B+S) and period of immersion (months).

Source of variation	DF	SS	MS	(F)	p value
Technique (tech)	2	174.08	87.04	0.18	0.84
Residue I	15	7245.12	483.01		
Main block	17	7419.20	570.05		
Period (per)	8	3001.51	375.19	5.39	**0.00(*)**
Interaction per x tech	16	629.80	39.36	0.57	0.90
Residue II	120	8347.38	69.56		
Sub-block	144	11978.69	484.11		
Total	161	19397.89	1054.16		

* Means significant difference at the level of 5%.

a significant reduction in the gap width without any significant difference in mechanical properties.

The process of water sorption depends on the composition of the composite (volume of filler, type of monomer of the resin matrix[11, 13]), volume of restoration and the cavity configuration, this last one related to the area of composite exposed to the oral fluids[7, 8]. Some authors have found that, the more hydrophobic the resin polymer and the more the percentage of such polymer in the resin matrix, the lower the water sorption[7, 8]. Besides, the larger the molecule size, its solubilization is more difficult[9].

It could be expected, in the present work, a superior increase of adhesive interface integrity over time for the B+S filling technique, since the flowable, self-cured resin composite placed between the Tetric Ceram and cavity walls has more percentage of organic matrix than this last one. However, it was not verified a significant influence of time immersion in water on the gap width, or a regular increase or decrease of the percentage of interface free of gaps over time for any filling technique.

The relatively high cavity configuration factor (around 3.5) and the use of hybrid light-cured composite chosen by the present work, with a high percentage of inorganic filler volume, may have collaborated with the limitation of water sorption during the initial 48 h, before the first analysis of the interface. However, after the restoration had been sliced, the contact between the restorative material and the water increased, which should have facilitated the water sorption since then. A feasible explanation for this fact can be the lack of uniformity of composite expansion, which may has occurred in a direction that did not contributed for the gaps closure. Another phenomenon that could explain the absence of significant influence of time immersion is the size difference between the polymer chains and the water molecule. As the water molecule

is much smaller than the resin matrix molecules size, it may be possible the diffusion of the water into the spaces among these molecules, increasing the mass of composite (as it has been found by some researches that immersed the composite in fluids[2, 4, 11, 14]), but without an increase of its volume.

Some authors reported an increase in the amount of interface with gaps over time of storage in water[15, 16]. Some of them[16] explain this occurrence as the effect of degradation of the polymeric structure, with hydrolytic degeneration of resin composite as the water diffuses along the adhesive interface.

While analyzing the chemical composition of substances found in the tooth-restoration interface over time, a study[17] reported that they were resultant of a degradation process of the composite structure. This study verified that the hygroscopic expansion was facilitated in regions with an incomplete cure of the composite and in gaps with the presence of oxygen. The same work observed that initial gaps may be closed or have their width reduced by the expansion of restorative material, but with hydrolytic degradation, more gaps are generated over time. This can also be a feasible explanation for the absence of a regular sequence of increase or decrease in the interface integrity over the months of measurements in the present work (only the fifth month presented a significant difference in the percentage of interface free of gaps).

CONCLUSION

It was not verified a significant influence of storage time in water on the integrity of tooth-restoration interface for any of three filling techniques. Some possible explanations for this occurrence are: (1) gain of mass without significant increase in the volume, due to the size difference between resin molecules and water molecule; (2) the expansion of restorative material in directions which did not contributed to the gaps closure; (3) simultaneous occurrence of hygroscopic expansion and hydrolytic degradation processes of the polymeric matrix over time immersion.

ACKNOWLEDGEMENTS

The authors would like to thank the financial support given by FAPESP. They would also like to thank Antonio Carlos Lascala and Sílvio Peixoto Soares, from the Department of Dental Materials, School of Dentistry, University of São Paulo (São Paulo/SP, Brazil) by technical support.

REFERENCES

1. Kemp-Scholte CMDavidson CL. Overhang of Class V composite resin restorations from hygroscopic expansion. Quintessence Int. 1989;20(8):551-3.

2. Berger SB, Palialol AR, Cavalli V, Giannini M. Characterization of water sorption, solubility and filler

particles of light-cured composite resins. Braz Dent J. 2009;20(4):314-8.

3. Sideridou I, Achilias DS, Spyroudi C, Karabela M. Water sorption characteristics of light-cured dental resins and composites based on Bis-EMA/PCDMA. Biomaterials. 2004;25(2):367-76.

4. Zhang YXu J. Effect of immersion in various media on the sorption, solubility, elution of unreacted monomers, and flexural properties of two model dental composite compositions. J Mater Sci Mater Med. 2008;19(6): 2477-83.

5. Loguercio AD, Reis A, Ballester RY. Polymerization shrinkage: effects of constraint and filling technique in composite restorations. Dent Mater. 2004;20(3):236-43.

6. Loguercio AD, Reis A, Schroeder M, Balducci I, Versluis A, Ballester RY. Polymerization shrinkage: effects of boundary conditions and filling technique of resin composite restorations. J Dent. 2004;32(6):459-70.

7. Feilzer AJ, de Gee AJ, Davidson CL. Relaxation of polymerization contraction shear stress by hygroscopic expansion. J Dent Res. 1990;69(1):36-9.

8. Carvalho RM, Pereira JC, Yoshiyama M, Pashley DH. A review of polymerization contraction: the influence of stress development versus stress relief. Oper Dent. 1996;21(1):17-24.

9. Ortengren U, Wellendorf H, Karlsson S, Ruyter IE. Water sorption and solubility of dental composites and identification of monomers released in an aqueous environment. J Oral Rehabil. 2001;28(12):1106-15.

10. Pearson GJ. Long term water sorption and solubility of composite filling materials. J Dent. 1979;7(1):64-8.

11. Ferracane JL. Hygroscopic and hydrolytic effects in dental polymer networks. Dent Mater. 2006;22(3): 211-22.

12. Munksgaard EC. Changes in expansion and mechanical strength during water storage of a traditional and three modified resin composites. Acta Odontol Scand. 2002;60(4):203-7.

13. Archegas LR, Caldas DB, Rached RN, Vieira S, Souza EM. Sorption and solubility of composites cured with quartz-tungsten halogen and light emitting diode light-curing units. J Contemp Dent Pract. 2008;9(2):73-80.

14. Drummond JL. Degradation, fatigue, and failure of resin dental composite materials. J Dent Res. 2008;87(8):710-9.

15. Mehl A, Kunzelmann KH, Hickel R. Quantitative und qualitative randspaltanalyse zervikaler füllungen. Dtsch Zahnäztl Z 1994;49:815-18.

16. Haller B, Hofmann N, Klaiber B, Pfannkuch A. Beständigkeit des komposit-dentin-verbundes bei künstlicher alterrung. Dtsch Zahnäztl Z. 1993;48:100-104.

17. Thonemann BM, Federlin M, Schmalz G, Hiller KA. SEM analysis of marginal expansion and gap formation in Class II composite restorations. Dent Mater. 1997;13(3):192-7.