

Mechanical and bonding properties of different combinations of nanohybrid and bulk-fill composites

Beatriz A. Ferrari, María M. Asueta, Laura G. Fusaro, Andrea E. Kaplan

Universidad de Buenos Aires, Facultad de Odontología, Cátedra Materiales Dentales, Buenos Aires, Argentina

ABSTRACT

The aim of this research was to determine compressive and shear bond strength of blocks prepared with bulk-fill and nanofill composite resin combinations. Materials used were Filtek Bulk Fill (FBF) and Z350 (both 3M-ESPE) and Surefil SDR flow (SFF) - Dentsply. To determine shear bond strength, cylindrical specimens 10 mm thick were prepared with composite consisting of thicknesses of 6 mm of one material and 4 mm of the other, in the following combinations: G1: FBF- FBF; G2: Z350-Z350, G3: FBF-Z350, G4: Z350-SFF and G5: SFF-SFF. Materials were cured using a 1100 mw/cm² light for 20 seconds for each layer. Samples were stored for 24 hours at 37 °C in distilled water and shear bond strength was determined. To assess compressive strength, cylindrical samples 4 mm diameter and 6 mm thick consisting of 4 mm + 2 mm were used in the same combinations as described above, stored in distilled water at 37 °C for 24 hours, after which compressive strength was determined. Both tests were performed with a Universal testing machine at a cross head speed of 1 mm/min. Results

were analyzed with ANOVA and Tukey's test.

Means and standard deviations in MPa for each group were the following: Shear bond strength: G1: 435.87 (65.86), G2: 233.6 (108.15), G3: 279.2 (22.05), G4: 449.1 (109.35) and G5: 196.6 (51.16). Compressive strength: G1: 160.07(4.27), G2: 149.49 (14.06), G3: 156.10 (29.99), G4: 199-30(39.28), G5: 171.23 (28.71). Evaluation with ANOVA showed no significant differences among combinations for compressive strength ($p > 0.05$) and significant differences for bond strength ($p < 0.05$). Tukey's test showed three homogeneous groups.

Under these experimental conditions, it can be concluded that the study combinations have adequate mechanical behavior, equivalent to materials used individually. However, shear bond strength was affected by the combinations analyzed.

Received: April 2021; Accepted: August 2021.

Keywords: composite resins - shear strength - compressive strength.

Propiedades mecánicas y adhesión entre diferentes combinaciones composite Nanohíbrido- Bulkfill

RESUMEN

El objetivo de este trabajo fue determinar la resistencia compresiva (RC) y la resistencia adhesiva al corte (RAC) en bloques preparados con combinaciones de composites bulk-fill y nanoparticulados. Los materiales usados fueron Filtek Bulk Fill (FBF) y Z350 (ambos de 3M-ESPE) y Surefil SDR flow (SFF) - Dentsply. Para medir la RAC, se prepararon probetas cilíndricas de 10 mm de espesor consistentes en 6 mm de un material y 4 mm del otro con las siguientes combinaciones: G1: FBF- FBF; G2: Z350-Z350, G3: FBF-Z350, G4: Z350-SFF y G5: SFF-SFF. Se curaron a 1100 mw/cm² durante 20 segundos cada capa. Se conservaron 24 horas a 37 °C en agua destilada antes de determinar la RAC. Para medir la RC se prepararon probetas de 4 mm de diámetro y 6 mm de espesor (4 mm + 2 mm de cada material), con las mismas combinaciones. Se conservaron en agua destilada a 37 °C durante 24 horas y se midió la RC. Ambos ensayos se realizaron con una máquina universal para ensayos mecánicos a 1 mm/min de velocidad de desplazamiento de cabe-

zal. Los resultados se evaluaron con ANOVA y prueba de Tukey. Las medias y desviaciones estándar (MPa) para cada grupo fueron: RAC: G1: 435.87 (65.86), G2: 233.6 (108.15), G3: 279.2 (22.05), G4: 449.1 (109.35) y G5: 196.6 (51.16). RC: G1: 160.07(4.27), G2: 149.49 (14.06), G3: 156.10 (29.99), G4: 199-30(39.28), G5: 171.23 (28.71). ANOVA no mostró diferencias estadísticamente significativas para RC ($p > 0.05$) y diferencias significativas para RAC ($p < 0.05$). La prueba de Tukey mostró tres grupos homogéneos.

En las condiciones experimentales de este trabajo puede concluirse que las combinaciones evaluadas tienen un comportamiento mecánico adecuado equivalente al de los materiales individuales. Sin embargo, la adhesión entre materiales se vio afectada por las combinaciones realizadas.

Palabras clave: resinas compuestas - resistencia al corte - resistencia compresiva.

INTRODUCTION

Composites have become the material of choice in restorative dentistry because of their adequate mechanical behavior, aesthetic properties and above all, conservation of healthy tissue. According to Ferracane et al.¹, one of the deficiencies of conventional composites with regard to the polymerization reaction is that the volume of the material contracts by about 3%². This contraction is transmitted to the interface between dental tissue and restorative material and may cause marginal filtration, secondary caries, loss of the restoration, cuspal deflection³, and enamel micro-cracks⁴, leading to postoperative sensitivity, usually during chewing. Various techniques and composites have been developed to minimize polymerization contraction and its clinical effects. The incremental technique is widely recommended for minimizing these problems. Placing layers 2 millimeters thick enables light to reach deeper zones, thereby achieving an adequate degree of conversion. However, this technique has some disadvantages such as inclusion of air bubbles, increased risk of contamination between layers, and longer working time^{5,6}. Most improvements have focused on modifying the filling, which has improved mechanical properties, mainly resistance to wear. Despite this progress, average lifespan of these composite restorations was found to be only 10 years⁷. Initially, composite matrix was exclusively based on the chemistry of methacrylate, more specifically BisGMA, TEGDMA, BisEMA and UDMA, and in the past 20 years, approximately, alternative monomers have been developed with the aim of reducing polymerization contraction and stress, emphasizing the association between the development of stress and the formation of gaps between the restoration and the tooth structure⁸. One alternative attempted was to apply ring opening polymerization or monomers of very high molecular weight. In the former, the only material developed was Filtek LS (3M), based on the silorane chemistry. Both strategies were successful in reducing the contraction coefficient to 1%⁹ and above all, reducing polymerization stress, according to *in vitro* assessments¹⁰. Although there are a few available clinical trials, results reported are contradictory. Popoff et al. found similar clinical behavior after one year with silorane-based and dimethacrylate-based resins in restorations¹¹. However, they note that studies should have longer

follow-up periods. Gonçalves et al. conducted an 18-month double blind, randomized study, finding that marginal integrity was worse in restorations with silorane-based composites than in those with dimethacrylate, finding no benefit in using this kind of composites in the restoration of Class II lesions¹².

More recently, greater importance has been assigned to improving resistance to breakdown in the oral medium –including hydrolysis of ester groups present in the methacrylates– caused by saliva and bacterial enzymes, and to preventing biofilm formation on the surface and interface of a composite restoration¹³. The most recent strategy has been to develop materials requiring fewer steps in their protocol for use, such as bulk-fill and self-adhesive composites. Bulk-fill composites have become increasingly popular for general practices. Although they can be classified in several different ways, they are best identified according to in-depth polymerization capacity, since they may have high or low viscosity, higher and lower ceramic load, and a great variety of mechanical properties¹⁴. The term bulk-fill has thus been used by manufacturers to refer to composites that can be inserted and polymerized in a single block of 4-5 mm. Some examples of the modifications made to conventional composite are use of monomers with high molecular weight such as AUDMA (Aromatic urethane dimethacrylate) and monomers known as AFM (addition-fragmentation monomers)¹⁵. Regarding ceramic filling, the percentage in volume was reduced in both bulk-fill flowable composites and bulk-fill composites with regular viscosity, with the percentage of filling in volume being even lower in the former. This reduction in percentage of filling reduces the difference in the refraction index between the matrix and the ceramic filling, which increases translucency for the material, enabling in-depth curing¹⁶. Moreover, the elastic modulus of these materials is lower than in conventional composites, and the rigidity of bulk-fill flowable composites is even lower than that of regular viscosity bulk-fill composites. This is why bulk-fill flowable composite manufacturers recommend placing an additional 2 mm layer of a conventional composite in zones exposed to greater stress¹⁷. The main properties that have

been studied are degree of conversion and depth of cure¹⁸. However, no papers were found studying bond strength between composite Z350 3M ESPE –Filtek Bulk Fill 3M ESPE and Z350 3M ESPE–Surefil SDR flowable or the mechanical properties of these combinations. Thus, the aim of this study was to determine shear bond strength and compressive strength of different combinations of bulk-fill composites and conventional composites.

MATERIALS AND METHODS

The following materials were used: Filtek Bulk Fill (FBF) and Z350 (both 3M-ESPE), and Surefil SDR flow (SFF) (Dentsply). To determine shear bond strength, test specimens (n=3) 10 mm thick and 4 mm in diameter were prepared in cylindrical molds. This 10 mm thickness consisted of 6 mm of one material and 4 mm of another in the following combinations: G1: FBF- FBF; G2: Z350-Z350, G3: FBF-Z350, G4: Z350-SFF and G5: SFF-SFF. They were polymerized using a Colt lux LED light-curing unit (Coltene) with intensity 1100 mw/cm² for 20 seconds per layer, following the thickness is recommended by the manufacturers in their respective instructions. An extra-fine marker (Edding 1880 Drawliner 0.1) was used to draw a line between the two parts. Specimens were embedded in self-curing acrylic resin cylinders (Subiton SL, Argentina) to make them easier to handle during mechanical assays. Specimens were stored for 24 hours at 37 °C in distilled water. The blocks prepared were placed on a support and load was applied at the level of the interface between the two materials with a metal needle until fracture occurred. Bond strength was determined using a universal testing machine for mechanical assays (1100. Instron Corporation) at a crosshead speed of 1 mm/minute. Fig. 1 shows the system used for determining shear bond strength. To determine compressive strength, cylindrical specimens (n=4) 4 mm in diameter and 6 mm thick were prepared. These 6 mm corresponded to 4 mm + 2 mm of the same combinations as described above. Specimens were stored in distilled water at 37 °C for 24 hours. Compressive strength was determined by placing an axial load using the same machine at the same crosshead speed as described for bond strength. Results were analyzed using ANOVA and Tukey's test with significance level p<0.05.

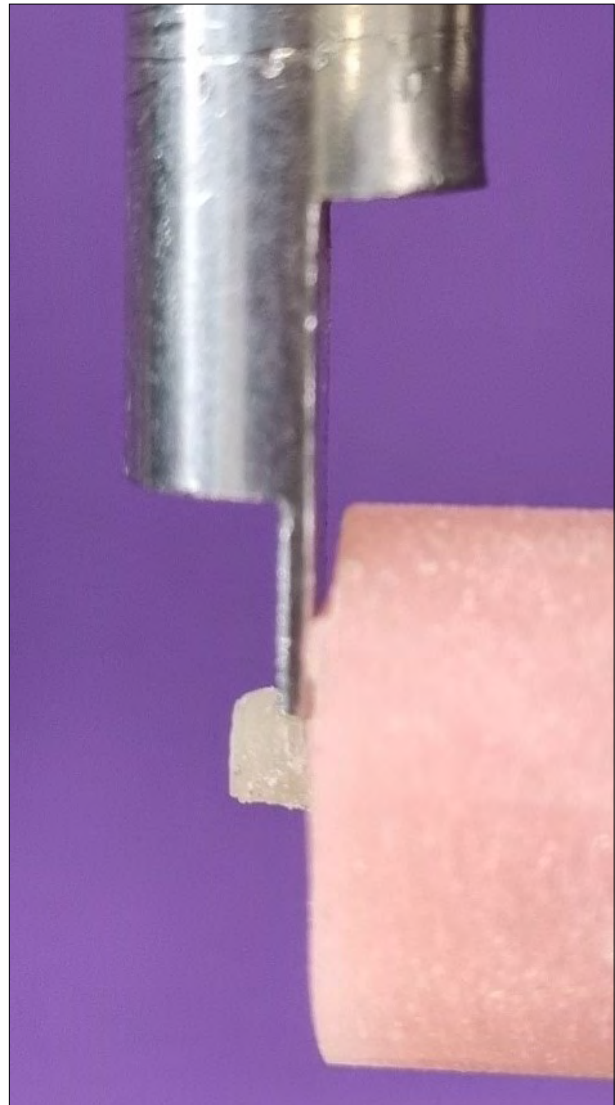


Fig. 1: Figure showing measurement of bond strength between parts made of different composites.

RESULTS

Mean and standard deviation in MPa for each group were the following: Bond strength: G1: 435.87 (65.86), G2: 233.6 (108.15), G3: 279.2 (22.05), G4: 449.1 (109.35) and G5: 196.6 (51.16); Compressive strength: G1: 138.61 (18.92), G2: 156.06 (9.71), G3: 167.18 (35.89), G4: 199.3 (39.28) and G5: 171.23 (28.71). Figs. 2 and 3 show mean and standard deviation for each. Evaluation with ANOVA showed no significant difference between combinations for compressive strength (p>0.05) but did show significant differences in bond strength (p<0.05). Tukey's Test showed three homogeneous groups for comparison of shear bond strength, suggesting statistically significant difference between G4 and G5 (between combination G4: Z350-SFF and

combination G5: SFF-SFF. This is shown in Fig. 2 with the same letters indicating homogeneous groups.

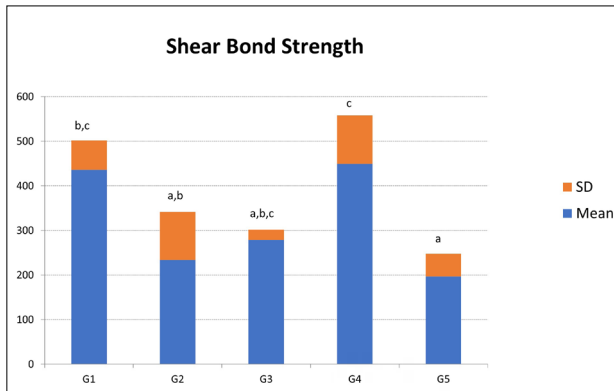


Fig. 2: Shear bond strength results. Mean (MPa) and standard deviation are shown for each group. Letters above the bars show results of comparisons using Tukey's test (the same letter in different groups indicates that the difference between them is not statistically significant).

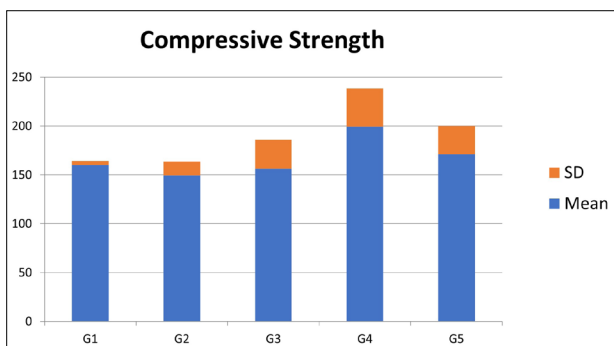


Fig. 3: Compressive strength results. Mean (MPa) and standard deviation are shown for each group.

DISCUSSION

The introduction on the market of bulk-fill composites raised two major questions: one regarding their ability to be photopolymerized adequately at the depths stated by the manufacturer, and the other regarding whether their mechanical properties might be deficient. As mentioned in the review by Camila Nuñez et al. regarding the advantage of shorter operative times, it would be interesting to evaluate whether the single block technique really saves clinical time¹⁹. Tiba et al. report that using bulk-fill flowable composites, which require an additional 2 mm occlusal layer of conventional composite, necessarily involves filling cavities with at least two increments, with different composites, which may not differ much from the operative times required for

4 mm cavities filled with conventional composites²⁰. As there is some evidence of the lower mechanical properties in bulk-fill flowable and regular viscosity composites, dentists often choose to use them as a base and cover them with conventional composites. The in vitro studies evaluating marginal seal have found results which are comparable to those using conventional composites. However, the same is not true for the evaluation of their mechanical properties^{21,22}. Esteves Lins et al. evaluated the mechanical properties of different composites, finding results in which they were unable to associate composite type –in terms of form of insertion– with compressive strength, having found similar values with no statistically significant difference compared to those inserted as a block²³.

This is consistent with the behavior observed in the current study, where there was no significant difference between the different combinations. This may also be attributed to the fact that this type of material does not differ from conventional materials in ceramic load only, but in a combination of factors²⁴. De Assis et al. reached similar conclusions²⁵.

According to Haughen et. al., the composite FBF presents a specific monomer with high molecular weight without free hydroxy groups in order not to increase its viscosity, in addition to lower ceramic content. Although this should provide better light penetration, some of the particles are silica or silanized zirconia, which have a high refractive index, leading to lower light transmission than in other bulk fill composites such as SFF, which could cause a lower degree of conversion¹⁴. In our study, group 4 (Z350- SFF) had the highest mean bond strength, even though different materials had been combined. This could be associated to the lower degree of conversion of monomers in the organic matrix of Z350²⁶, which may leave a larger number of double bonds available for bonding to SFF. In turn, SFF has the lowest percentage of ceramic filling in volume (45 vol%), which may also contribute to providing a larger quantity of available monomers for bonding between the two materials.

Under the experimental conditions in this study, it can be concluded that the combinations of materials evaluated have similar mechanical behavior. However, bonding between them was affected in the combinations.

DECLARATION OF CONFLICTING INTERESTS:

The authors declare no potential conflicts of interest regarding the research, authorship, and/or publication of this article.

FUNDING

This study was financed by Subsidy 20020160100037BA from the Scientific Program of Buenos Aires University's Secretariat of Science and Technology.

CORRESPONDENCE

Dr. Andrea E. Kaplan
Cátedra de Materiales Dentales
Facultad de Odontología, UBA
Marcelo T. de Alvear 2142
Ciudad Autónoma de Buenos Aires
Argentina
Andrea.kaplan@odontologia.uba.ar

REFERENCES

- Ferracane JL. Resin composite-State of the art. *Dent Mater* 2011;27:29-38.
- Kim YJ, Kim RJ, Ferracane J, Lee IB. Influence of the Compliance and Layering Method on the Wall Deflection of Simulated Cavities in Bulk-fill Composite Restoration. *Oper Dent* 2016;41:183-194.
- Kim RJ, Kim YJ, Choi NS, Lee IB. Polymerization shrinkage, modulus, and shrinkage stress related to tooth-restoration interfacial debonding in bulk-fill composites. *J Dent* 2015; 43:430-439.
- Hannig M, Friedrichs C. Comparative in vivo and in vitro investigation of interfacial bond variability. *Oper Dent* 2001;26:3-11.
- Sakaguchi RL, Douglas WH, Peters MC. Curing light performance and polymerization of composite restorative materials. *J Dent* 1992;20:183-188.
- Pilo R, Oelgiesser D, Cardash HS. A survey of output intensity and potential for depth of cure among light-curing units in clinical use. *J Dent* 1999;27:235-241.
- Demarco FF, Corrêa MB, Cenci MS, Moraes RR et al. Longevity of posterior composite restorations: not only a matter of materials. *Dent Mater* 2012;28:87-101.
- Feng L, Suh BI, Shortall AC. Formation of gaps at the filler-resin interface induced by polymerization contraction stress: Gaps at the interface. *Dent Mater* 2010;26:719-729.
- Weinmann W, Thalacker C, Guggenberger R. Siloranes In Dental composites. *Dent Mater* 2005;21:68-74.
- Boaro LC, Gonçalves F, Guimarães TC, Ferracane JL et al. Polymerization stress, shrinkage and elastic modulus of current low-shrinkage restorative composites. *Dent Mater* 2010;26:1144-1450.
- Popoff DA, Santa Rosa TT, Ferreira RC, Magalhães CS et al. Repair of dimethacrylate-based composite restorations by a silorane-based composite: a one-year randomized clinical trial. *Oper Dent* 2012;37:E1-10. doi: 10.2341/11-121-C.
- Gonçalves FS, Leal CD, Bueno AC, Freitas AB et al. A double-blind randomized clinical trial of a silorane-based resin composite in class 2 restorations: 18-month follow-up. *Am J Dent* 2013;26:93-98.
- Wu J, Weir MD, Melo MA, Xu HH. Development of novel self-healing and antibacterial dental composite containing calcium phosphate nanoparticles. *J Dent* 2015;43:317-326.
- Haugen HJ, Marovic D, Par M, Thieu MKL et al. Bulk Fill Composites Have Similar Performance to Conventional Dental Composites. *Int J Mol Sci* 2020;20:5136. doi: 10.3390/ijms21145136.
- Fugolin APP, Pfeifer CS. New Resins for Dental Composites. *J Dent Res* 2017;96:1085-1091.
- Czasch P, Ilie N. In vitro comparison of mechanical properties and degree of cure of bulk fill composites. *Clin Oral Investig* 2013;17:227-235.
- Ilie N, Bucuta S, Draenert M. Bulk-fill resin-based composites: an in vitro assessment of their mechanical performance. *Oper Dent* 2013;38:618-625.
- Alrahlah A, Silikas N, Watts DC. Post-cure depth of cure of bulk fill dental resin-composites. *Dent Mater* 2014; 30:149-154.
- Maas MS, Alania Y, Natale LC, Rodrigues MC et al. Trends in restorative composites research: what is in the future? *Braz Oral Res* 2017;28;31:e55 doi: 10.1590/1807-3107BOR-2017.vol31.0055.
- Tiba A, Zeller GG, Estrich CG, Hong A. A laboratory evaluation of bulk-fill versus traditional multi-increment-fill resin-based composites. *J Am Dent Assoc* 2013;144:1182-1183.
- Roggendorf MJ, Krämer N, Appelt A, Naumann M et al. Marginal quality of flowable 4-mm base vs. conventionally layered resin composite. *J Dent* 2011;39:643-647.
- Campos EA, Ardu S, Lefever D, Jassé FF et al. Marginal adaptation of class II cavities restored with bulk-fill composites. *J Dent* 2014;42:575-581.
- Lins RBE, Aristilde S, Osório JH, Cordeiro CMB et al. Biomechanical behaviour of bulk-fill resin composites in class II restorations. *J Mech Behav Biomed Mater* 2019;98:255-261.
- Leprince JG, Palin WM, Vanacker J, Sabbagh J et al. Physico-mechanical characteristics of commercially available bulk-fill composites. *J Dent* 2014;42:993-1000.
- de Assis FS, Lima SN, Tonetto MR, Bhandi SH et al. Evaluation of Bond Strength, Marginal Integrity, and Fracture Strength of Bulk- vs Incrementally-filled Restorations. *J Adhes Dent* 2016;18:317-323.
- Salem HN, Hefnawy SM, Nagi SM. Degree of Conversion and Polymerization Shrinkage of Low Shrinkage Bulk-Fill Resin Composites. *Contemp Clin Dent* 2019;10:465-470.