Degree of Conversion and Mechanical Properties of a Commercial Composite with an Advanced Polymerization System

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ABSTRACT

Advanced Polymerization System (APS) technology in a commercial composite resin enables reduction of the concentration of camphorquinone without altering composite physicochemical properties. **Aim:** The aim of this study was to evaluate the degree of conversion and mechanical properties of a commercial composite with an advanced polymerization system (APS) and compare it to other composites that do not use this system. **Materials and Method:** Five groups were analyzed. Group 1 (VT: Vittra APS - FGM); G2 (AU: Aura – SDI); G3 (ES: Quick Sigma Stelite – TOKOYAMA); G4 (FZ: Filtek Z350 XT – 3M ESPE); G5 (OP: Opallis -FGM). Degree of conversion (DC, n=3) was analyzed immediately and after 24h by analysis with FTIR spectroscopy. For Knoop hardness (KHN, n=3), 5 indentations were made at the top and bottom of specimens 2 mm thick. Flexural strength (FS, n=10) was determined by the three-point method in a universal testing machine. Polymerization stress (PS) was determined by light-curing the material (1.0 mm high) between polymethylmethacrylate rods in a universal testing machine. Light curing was performed with a Valo Cordless LED (1,000 mW/cm2 x 20 s: 20J). The results were analyzed using ANOVA and complemented by Tukey's test (α=0.05). **Results:** The highest DC values (immediate and 24h after) were observed for VT and OP resins, followed by FZ, AU and ES. FZ (top and bottom) had the highest KHN values, similar to VT top. AU, ES, OP and VT had statistically different KHN between their top and bottom surfaces. The highest RF values were observed for FZ, followed by OP/VT, ES and AU. The highest TP values were observed for FZ, OP and VT. **Conclusion:** The Vittra APS resin with a new polymerization system presents satisfactory performance for the parameters evaluated.

Keywords: composite resin - polymerization - camphorquinone.

Avaliação do grau de conversão e das propriedades mecânicas de um compósito comercial com sistema avançado de polimerização

RESUMO

A tecnologia Advanced Polymerization System (APS) presente em uma resina composta comercial permite reduzir a concentração de canforoquinona sem alterar as propriedades físico-químicas do compósito. **Objetivo:** o objetivo deste estudo foi avaliar o grau de conversão e as propriedades mecânicas de um compósito comercial com sistema avançado de polimerização (SAP) e compará-lo com outros compósitos que não utilizam esse sistema. **Materiais e Método:** cinco grupos foram analisados. Grupo 1 (VT: Vittra APS - FGM); G2 (AU: Aura – SDI); G3 (ES: Quick Sigma Stelite – TOKOYAMA); G4 (FZ: Filtek Z350 XT – 3M ESPE); G5 (OP: Opallis -FGM). O grau de conversão (GC, n=3) foi analisado imediatamente e após 24h através da análise com espectroscopia FTIR; para dureza Knoop (DK, n=3), foram feitas 5 indentações no topo e na base de corpos de prova de 2 mm de espessura; para determinar a resistência à flexão (RF, n=10), o método de três pontos foi realizado em uma máquina universal de ensaios; a tensão de polimerização (TP) foi determinada pela fotopolimerização do material (1,0 mm de altura) entre hastes de polimetilmetacrilato em uma máquina de teste universal. A fotopolimerização foi realizada com um Valo Cordless LED (1.000 mW/cm² x 20 s: 20J). Os resultados foram analisados por ANOVA e complementados pelo teste de Tukey (α=0,05). **Resultados:** os maiores valores de GC (imediato e 24h após) foram observados para as resinas VT e OP, seguidas de FZ, AU e ES. A resina FZ (superior e inferior) apresentou os maiores valores de DK, semelhante ao VT superior. As resinas AU, ES, OP e VT apresentaram DK estatisticamente diferente entre suas superfícies de topo e base. Os maiores valores de RF foram observados para FZ, seguido de OP/VT, ES e AU. Os maiores valores de TP foram observados para FZ, OP e VT. **Conclusão:** com base nos resultados, pode-se concluir que a resina Vittra APS com um novo sistema de polimerização apresenta desempenho satisfatório para os parâmetros avaliados.

Palavras-chave: resina composta - polimerização - canforoquinona.

INTRODUCTION

Progress in restorative Dentistry has led to composite resins being one of the most intensively studied materials, with the aim of improving their mechanical and optical properties[1].

Over the years, in addition to changes in the composition of resins, such as the incorporation of new monomeric formulations[2], there have been changes in the photoactivation systems. Composite resins activated by visible light initiate the polymerization process by absorbing light from a photoinitiator, which, once activated, reacts with a reducing agent to produce free radicals such as camphorquinone (CQ)[3]. The methacrylate monomers then polymerize, forming a cross-linked polymer matrix, with CQ being the most frequently used photoinitiator for light-curing resin[4]. A combination of the photoinitiator/co-initiator system must be used, usually CQ and a tertiary amine, respectively[5]. Despite being the most frequently used photoinitiator, CQ has some negative aspects such as toxicity and low polymerization efficiency[5]. Furthermore, it is a solid yellow compound and, even in small amounts, can lead to an undesirable color change. Another disadvantage is that the α-diketone group derived from CQ has an absorption peak of 468 nm in the visible range of light from 400 to 500 nm, causing rapid photopolymerization under ambient light (fluorescent and dental lamps), and resulting in short therapy operating time[5].

Higher amounts of CQ promote rapid generation of large amounts of free radicals. However, this can originate polymers with lower molecular weight and reduced ability to form a suitable polymeric network. On the other hand, if the concentration of photoinitiators is too low, an inadequately polymerized resin composite may result in altered mechanical properties, and the durability of the restoration may be compromised. Therefore, the concentration and proportion of photoinitiators and co-initiators that guarantee satisfactory polymerization behavior is of essential importance[5,6]. The FGM manufacturer claims that it developed the Advanced Polymerization System (APS), which enhances the components and allows the reduction of camphorquinone concentration, in order to improve the properties of the materials it manufactures. Technically, the APS system uses a smaller amount of CQ associated with several photoinitiators that interact with each other and enhance polymerization capacity. According to the manufacturer, the system ensures a higher degree of conversion, longer handling time under ambient light, and better mechanical properties compared to conventional systems based on CQ.

In addition to new polymerization systems for composite resins, there is a current trend in the production of materials free from Bisphenol-A, a substance present in the composition of Bis-GMA monomer and related to harmful effects in the body caused by some monomers[7]. As a result, FGM launched the first Brazilian resin with an organic matrix free from Bis-GMA monomers, the Vittra APS composite resin, which contains nanospheroidal zirconia particles, and provides aesthetics due to its high polishing capacity.

In this context, the present study aimed to evaluate *in vitro* the degree of conversion and mechanical properties of Vittra APS composite resin against other composites available on the Brazilian market.

MATERIALS AND METHOD

Type of study and materials

This was an experimental *in vitro* study, in which the variables were the photoinitiator system and the composition of the composite resins studied. The materials used in the present study are described in Table 1. The color selected for all materials was A3 enamel.

Degree of Conversion (DC)

The degree of conversion (n=3) was obtained by Fourier transform near-infrared spectroscopy (FT-IR Vertex 70, Brüker Optik GmbH - United Kingdom). The reading parameters used were: 4 cm^{-1}, 32 scans in the range between 2000 and 100 cm^{-1}, and power of 100 mW.

A 1.0 mm thick sheet of condensation silicone (Optosil - Heraeus Kulzer) was made, pressed between two glass plates with spacers and insulated with liquid Vaseline. The silicones obtained were cut into rectangles measuring 3.0 x 2.0 cm and a perforation of 8.0 mm in diameter was made in the center of the silicone. Finally, each cutout was positioned in the center of glass slides.

The material was inserted into the silicone mold and pressed by another glass slide. The sets were stabilized by adhesive tapes and taken to the Vertex 70 spectrometer (Brüker Optik GmbH) to determine

Table 1. Materials used in this study (as described by the manufacturers)

Material and manufacturer	Composition	Photoinitiator system
Vittra APS, FGM Dental Products	TEGDMA, UDMA, photoinitiator composition [APS], silane, zirconia particles (200 nm), silica: 72% to 82% by weight, 52% to 60% by volume. Composite resin composed entirely of nanometric fillers (100 - 200 mm)	Camphorquinone + other photoinitiators
Aura, SDI	Limited information provided by the manufacturer	Limited information provided by the manufacturer
Estelite Sigma Quick, Tokuyama	Bis-GMA, TEGDMA. Silica-zirconia particles with an average size of 0.2μm (82% by weight and 71% by volume)	Radical Amplified Photopolymerization Technology (RAP).
Filtek Z350XT, 3M ESPE	TEGDMA, Bis-EMA, Bis-GMA, UDMA, PEGDMA. Non-agglomerated silica and zirconia particles (20 nm and 4-11 nm, respectively) and aggregated Silica/zirconia (0.6 – 10 μm). 72.5% by weight and 55.5% by volume	Camphorquinone
Opallis, FGM	Bis-GMA, Bis-EMA, TEGDMA, UDMA, co-initiator, silane. Barium aluminum silicate glass (0.5 μm) silanized (78.5% to 79.8% by weight and 57.0 to 58% by volume).	Camphorquinone

the unpolymerized spectrum. Baseline correction and curve normalization were performed using the OPUS program (Brüker Optics), increasing intensity and facilitating visualization. The peak heights of the 1610 and 1640 cm^{-1} bands were measured and recorded.

The specimens were removed from the RFS100/S equipment and light-cured with a Valo Cordless LED device (Ultradent - South Jordan, United States), with an active tip of 10 mm in diameter, for 20 seconds under an irradiance of 1,000 mW/cm^2, resulting in an energy density of 20 J/cm^2. A new reading was performed. The specimens were evaluated immediately and after 24 hours stored in a dry environment. To calculate the DC after light-curing, the values of peak heights of the bands of uncured and light-cured material were used.

Knoop hardness numbers (KHN)

Cylindrical specimens (n=3) 4.0 mm in height, 2.5 mm in width, and 2.5 mm in depth were made with the aid of a bipartite circular brass die, then positioned on a polyester strip fixed on a glass plate. The resin was inserted into the die and pressed with a transparent strip of polyester and glass slide, followed by photoactivation of the material, as described for degree of conversion.

Subsequently, the irradiated face of the specimen was polished in an EcoMetTM/AumetTM 300 polisher (Buehler - Lake Bluff, United States) under abundant irrigation with 1200 grit sandpaper for 2 minutes (20 N force; speed: 70 rpm). After 24 hours,

an ultrasonic bath was performed, followed by 5 indentations with load of 200 g for 20 seconds in a Shimadzu HMV-2 hardness tester (Tokyo, Japan), to determine the hardness values of the top and bottom surfaces (surface directly irradiated by light, and surface opposite to light irradiation, respectively).

Flexural Resistance (FR)

Specimens (n=10) were made in a bipartite steel die (10 mm long, 2 mm wide, 1 mm thick) supported by a transparent strip on a glass plate. After inserting the material, a transparent strip of polyester and a glass slide were positioned over the die to accommodate the material, followed by photoactivation, as described for degree of conversion. After disocclusion of the specimen, the excesses were removed with the aid of a scalpel blade number 11 (Solidor - Barueri, São Paulo) and the dimensions of the specimen were recorded.

The flexural strength tests were performed in a universal testing machine (model 5565, Instrom Corp - Canton, United States) with load cell of 1000 N, test speed of 0.5 mm/min and action of the incident force on the irradiated face. The distance between the supports was 8 mm, always checked between groups with a digital caliper.

Specimen breaking load values (in Newtons) and dimensions were recorded for calculation of maximum flexural strength in MPa.

Polymerization Stress (PS)

For the PS test, the method by Gonçalves et al.[7] was

used. Polymethylmethacrylate (PMMA) cylinders (Ø3mm) were sectioned into rods 13 and 28 mm long. For the 13 mm rods, one end was polished with 1200, 2000 and 4000 grit sequence sandpaper and felt with 1 µm alumina paste to allow the passage of blue light along the length of the rod at the time of photoactivation. The other faces of the other rods, both 13 and 28 mm long, were sprayed with aluminum oxide (250 µm) to improve the adhesion of the hydrophobic resin to the PMMA.

To prepare each specimen for the polymerization stress test, a layer of methyl methacrylate monomer (JET Self-polymerizing Acrylic, Clássico, Brazil) was applied to one of the sandblasted surfaces of the 28 mm rod and the sandblasted surface of the 13 mm rod to improve the wetting of the unfilled hydrophobic adhesive (Scotchbond Multiuso Plus, 3rd bottle, 3M ESPE, Brazil) applied later. After this last step, a jet of air 15 cm away from the surface of the rod was applied for 10 seconds with the aid of a triple syringe. The hydrophobic adhesive on both surfaces was individually light-cured for 20 seconds with a Valo Cordless LED device (Ultradent, South Jordan, United States).

The rods were manipulated such that contact with the treated surfaces was avoided, and then fixed in an Instron 5665 universal testing machine. The 28 mm rod was fixed in a grip connected to the load cell of the machine with the treated face facing downwards, and the 13 mm rod was positioned with the treated side facing up, in a device developed for the present test and coupled to the base of the testing machine. In order to evaluate the influence of the increment thickness on the polymerization stress, the distance (specimen height, h) between the rods was standardized at 1 mm, with 05 (n=5) being the number of specimens for each material.

After determining specimen height (distance between the rods) and recording it in the equipment memory, the load cell was moved away from the machine base and the material was inserted over the treated surface of the lower rod. The load cell was positioned according to the height parameter just memorized, so that the material could be pressed and in contact with both treated surfaces. After removing any excess material with the aid of a spatula, an extensometer (model 2360-101, Instron) was attached to the rods to keep the distance between them constant during the test.

The tip of the light-curing unit was positioned in the device slot and coupled to the base of the testing machine, directly in contact with the polished face of the base of the 13 mm rod. The tension developed during the polymerization of the materials was monitored for 15 minutes from the light-activation of the material. To determine the maximum rated polymerization stress (MPa), the maximum stress value recorded during the test (N) was divided by the cross-sectional area of each rod.

Statistical analysis
The data recoded in all tests were submitted to normality and homoscedasticity tests, then evaluated by analysis of variance (ANOVA) complemented by Tukey's test or evaluated only by the Kruskal-Wallis test ($\alpha < 0.05$).

RESULTS
Conversion Degree
Mean degree of conversion (%) for the different dental composites are shown in Figure 1. DC values immediately after curing ranged from 44.08% to 66.9%, while the 24-hour post-cure values ranged from 65.94% to 81.82%. For immediate cure, the highest to lowest mean rates of polymerization were: Vittra APS, Opallis, Filtek Z350XT, Estelite Sigma Quick and Aura. After 24 hours, DC% of Vittra APS and Opallis resins did not differ statistically, though their degree C = C conversion was higher than for Filtek Z350XT, Estelite Sigma Quick and Aura. DC after 24 h did not differ statistically between Estelite Sigma Quick and Aura. Thus, the advanced polymerization system technology in the Vittra APS resin may have influenced its performance.
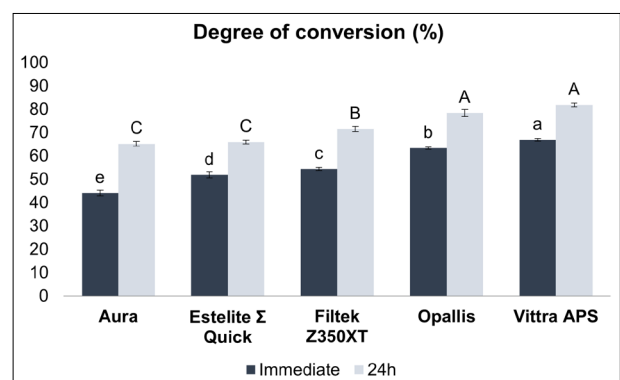

Fig. 1: Degree of conversion (%) of the resins evaluated immediately and 24 hours after light-curing.

Knoop Hardness (KHN)

The Knoop hardness results for each composite (the top of the specimen being the surface directly irradiated, and the bottom being the surface opposite to irradiation) are shown in Figure 2. Significant differences were found for bottom/top hardness among composites (p=0.010). Aura resin showed significantly lower values than the other materials. The Filtek Z350 XT resin (top and bottom) had the highest KHN values, with the bottom value being similar to the top value of Vittra APS. Despite Vittra's advanced polymerization technology, its KHN (in kilograms-force per square millimeter) at the top (44.15) was approximately double the value at the bottom (24.59).
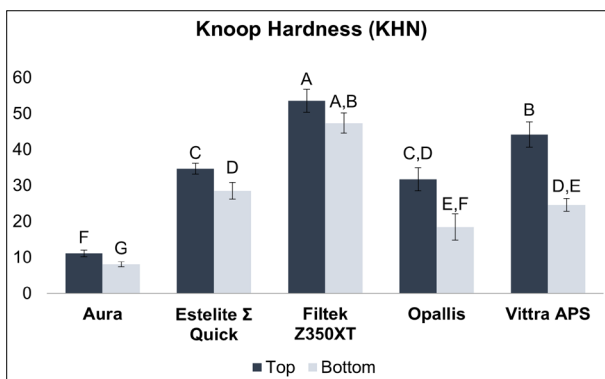

Fig. 2: Knoop hardness (KH) of the resins evaluated 24 hours after light-curing.

Flexural strength (Mpa)

The results the 3-point flexural strength (MPa) are shown in Figure 3. FS was statistically highest for Filtek Z350 XT, and lowest for Aura. Vittra APS and Opallis behaved similarly, with higher flexural strengths than Estelite, Sigma Quick and Aura.
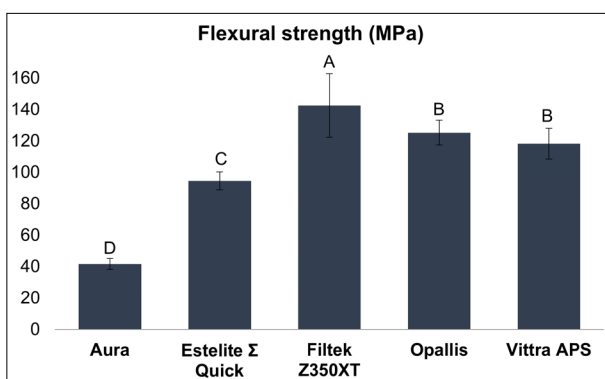

Fig. 3: Flexural strength (MPa) of the resins evaluated 24 hours after light-curing.

Polymerization Stress (Mpa)

The results of polymerization stress (MPa) are shown in Figure 4. The PS of the composite resins ranged from 2.68 MPa (Aura) to 3.84 MPa (Opallis). Polymerization shrinkage stress values differed significantly between some materials tested (p<0.001). Vittra APS showed higher polymerization stress than AURA (p=0.031). The highest values of polymerization stress were observed for Opallis, Filtek Z350 XT and Vittra APS, followed by Estelite Sigma Quick and Aura.
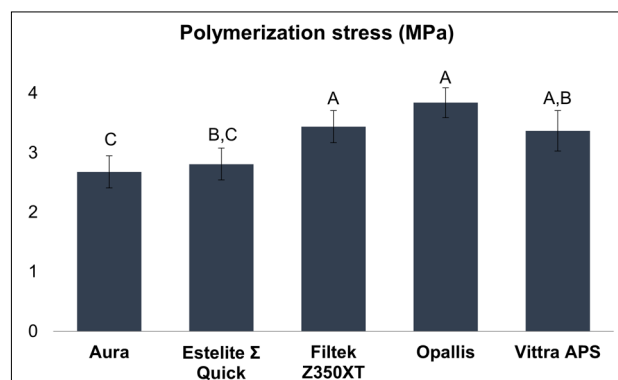

Fig. 4: Polymerization stress (MPa) of the evaluated resins.

DISCUSSION

Composite resin properties such as hardness and degree of conversion influence the clinical performance of restorations. The presence of non-converted double carbon links could increase susceptibility to degradation, reducing color stability and releasing residual monomers[8,9]. The present study observed that the DC of composite resins can vary from 34.7% to 77.1%, while the literature reports that this increase can be up to 36% in the first 24 hours[10]. Still, no material can completely convert into a polymer. In the present study, Vittra APS and Opallis resins, both from FGM, presented the highest DC, both immediately (statistically higher for Vittra APS) and 24 hours after polymerization (no statistical difference between the two). For Vittra APS, this can be explained by the presence of the APS system, which despite the low concentration of CQ, contains secondary photoinitiators not informed by the manufacturer and capable of guaranteeing high monomer conversion.

AURA (SDI) presented a statistically lower percentage than all the other resins tested in the immediate and 24 hours after readings. However, according to Alshali et al.[10], a minimum DC value

has not yet been established for adequate clinical performance. It should be noted that little information was made available by the manufacturer about this material. Many factors can affect polymerization efficiency, whether intrinsic (type and concentration of photoinitiator, matrix viscosity and optical properties) or extrinsic (light type and spectrum, irradiation parameters, curing mode, temperature and light tip positioning)[11]. This study used the same light source for all tests and resins, setting the same distance from the tip to the specimen and in the same work environment, in order to eliminate as far as possible any other variables that could affect the DC.

Despite being controversial, some studies have shown a correlation between DC and hardness[12,13], which can be explained by the fact that the strength of the material is determined not only by the inorganic fraction, but also by the creation of a dense, cross-linked polymeric network[12]. A base-to-top hardness ratio (B/T) ranging from 0.8 to 0.9 is an important criterion to verify the efficiency of polymerization in deeper areas in relation to the light source. Theoretically, the base surface hardness of a polymerized composite should be at least 80% (0.8) of the top surface hardness[6].

In the present work, AURA (SDI) had the lowest KHN hardness values (B/T). Opallis and Estelite Sigma Quick showed statistically similar mean KHN hardness values at the top. Hardness at the base of Opallis did not reach the desirable 80% B/T. Vittra hardness was higher at the top, compared to the aforementioned resins, and statistically similar to the Z350 XT resin base. Z350 XT top hardness was statistically significantly the highest among the tested resins.

A study on the effect of adding zirconia particles to a commercial composite (Vittra APS) reported that regardless of its concentration, zirconia increases hardness values compared to the control group in which the manufacturer provided the composition[14]. The presence of nano spheroidal zirconia particles combined with the expected dense polymeric network at the top of the specimens due to the presence of APS technology may account for the high hardness value compared to the base of the same material. In this case, the presence of the APS system does not seem to guarantee greater depth of polymerization.

Widely used in laboratory tests of dental composites,

flexural strength is one of the main parameters for assessing a material's resistance to fracture[14]. Flexural strength represents the stress experienced by a material at the time of its rupture and is expressed in megapascals. Z350 XT had the highest flexural strength, with statistical significance. Opallis and Vittra APS had intermediate and similar values, followed by Estelite and Aura. Aura had the lowest flexural strength among the tested resins.

According to ISO standard 4049, used to evaluate the flexural strength of restorative materials, the minimum acceptable value for composite resins is 100 Mpa[15]. Borges et al.[16] evaluated the flexural strength of 8 direct composite resins, which were microparticulate, microhybrids, hybrids and just a nanocluster, containing silica particles. This nanocluster presented the highest values for flexural strength, which were close to 150 Mpa. In the present study, the nanoparticulate resins also presented the highest values for flexural strength, with Z350 XT above 140 MPa, and Vittra APS with a value close to 120 MPa, which are values higher than those required by ISO 4049.

Borges et al.[16] report that increasing the amount of filler significantly improves all mechanical properties of composite resins. In our study, Vittra APS had a higher percentage of filler than the other composites tested, only lower than Opallis, though statistically similar. The size of the filler particles also affects the material's strength, that is, composites with nanometer-scale filler particles present greater resistance to fracture.

During polymerization, monomer molecules join to form a network of polymers. As the network is formed, the molecules approach each other so that new bonds can form[17]. This approach among molecules causes volume contraction, which can cause cracks, margins, pigmentation, microleakage, secondary caries, and postoperative sensitivity, in addition to causing tensions in the tooth-restoration interface. These tensions, in turn, can cause microfractures and failures[18]. The shrinkage stress is proportional and may be related to the volumetric shrinkage and the elastic modulus of the composites[18,3]. Other factors such as filler content, resin matrix composition and degree of monomeric conversion can also influence polymerization tension[19]. Despite the presence of the APS system in the Vittra resin, it did not present low polymerization stress, but had results like Filtek Z350 XT, Opallis

and Estelite. The lowest polymerization stress values were observed for Aura, possibly related to its low degree of monomeric conversion. Despite the limitations of the study, the resin with the APS system showed encouraging outcomes, both for aesthetic and mechanical properties.

CONCLUSION

In this study, Vittra APS resin, with a new polymerization system, presents satisfactory performance for the parameters evaluated when compared to the other materials tested. Despite having a high degree of conversion and polymerization stress like the other resins, it was inferior to Filtek Z350 XT in the Knoop hardness and flexural strength tests.

DECLARATION OF CONFLICTING INTERESTS

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

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