

**UNIVERSIDADE ESTADUAL DE CAMPINAS  
FACULDADE DE ODONTOLOGIA DE PIRACICABA**

**Virginia Bosquioli**

**PROPRIEDADES MECÂNICAS E RESISTÊNCIA DA UNIÃO DE RESINAS  
COMPOSTAS COM MATRIZ DE SILORANO OU COM NANOPARTÍCULAS**

Tese apresentada à Faculdade de Odontologia de Piracicaba, da Universidade Estadual de Campinas, para obtenção do Título de Doutor em Materiais Dentários.

**Orientador: Prof. Dr. Mário Alexandre Coelho Sinhoreti**

**ESTE EXEMPLAR CORRESPONDE À VERSÃO FINAL DA TESE  
DEFENDIDA PELA ALUNA VIRGÍNIA BOSQUIROLI, E ORIENTADA PELO PROF.DR. MÁRIO  
ALEXANDRE COELHO SINHORETI**

---

**Assinatura do Orientador**

PIRACICABA, 2011

FICHA CATALOGRÁFICA ELABORADA POR  
GARDÊNIA BENOSSI – CRB8/8644 - BIBLIOTECA DA  
FACULDADE DE ODONTOLOGIA DE PIRACICABA DA UNICAMP

**Bosquioli, Virginia, 1970-**  
B652p Propriedades mecânicas e resistência da união de resinas compostas com matriz de silorano ou com nanopartículas / Virginia Bosquioli. -- Piracicaba, SP : [s.n.], 2011.

Orientador: Mário Alexandre Coelho Sinhoreti.  
Tese (doutorado) – Universidade Estadual de Campinas, Faculdade de Odontologia de Piracicaba.

1. *Partículas inorgânicas.* 2. *Restauração.* 3. *Polímeros.* I. *Sinhoreti, Mário Alexandre Coelho.* II. *Universidade Estadual de Campinas. Faculdade de Odontologia de Piracicaba.* III. *Título.*

Informações para a Biblioteca Digital

**Título em Inglês:** *Mechanical properties and bond strength of composite resins with silorane matrix or nanofillers*

**Palavras-chave em Inglês:**

Inorganic particles

Restoration

Polymers

**Área de concentração:** Materiais Dentários

**Titulação:** Doutor em Materiais Dentários

**Banca examinadora:**

*Mário Alexandre Coelho Sinhoreti [Orientador]*

*Alysson Noriyuky Kajishima Konno*

*William Cunha Brandt*

*Rafael Leonardo Xediek Consani*

*Fernanda Miori Pascon*

**Data da defesa:** 26-07-2011

**Programa de Pós-Graduação:** Materiais Dentários



UNIVERSIDADE ESTADUAL DE CAMPINAS  
Faculdade de Odontologia de Piracicaba



A Comissão Julgadora dos trabalhos de Defesa de Tese de Doutorado, em sessão pública realizada em 26 de Julho de 2011, considerou a candidata VIRGINIA BOSQUIROLI aprovada.

Prof. Dr. MARIO ALEXANDRE COELHO SINHORETI

Prof. Dr. ALYSSON NORIYUKI KAJISHIMA KONNO

Prof. Dr. WILLIAM CUNHA BRANDT

Prof. Dr. RAFAEL LEONARDO XEDIEK CONSANI

Profa. Dra. FERNANDA MIORI PASCON

## **DEDICATÓRIAS**

**A Deus, porque sempre é luz para o meu caminho e por tudo o que tem proporcionado para o meu crescimento pessoal e profissional.**

*Pedi, e dar-se-vos-á; buscai, e encontrareis; batei, e abrir-se-vos-á.*

Mateus, 7:07

**Aos meus amados pais, Eugênia e Ivo, pelo exemplo de perseverança, amor e serenidade de sempre.**

**A toda minha família pelo incentivo neste desafio profissional.**

**Ao meu namorado Nereo pelo apoio, paciência e pelo exemplo de determinação que é para mim.**

**As minhas queridas amigas Adriane Paglia e Celita Zambonato por aguçarem a minha vontade em conhecer melhor e aperfeiçoar a minha prática na odontologia restauradora.**

## **AGRADECIMENTO ESPECIAL**

**Ao Prof. Dr. Mário Alexandre Coelho Sinhoreti,**

Agradeço por todo conhecimento transmitido de forma detalhada e sempre muito embasada cientificamente e que tanto acrescentou para minha carreira docente. Considero para minha formação como futura orientadora em pesquisa o seu exemplo de caráter, humildade e tranqüilidade no trato com todos os seus alunos e orientados, principalmente nos momentos mais difíceis.

**Muito Obrigada!**

***“Mais do que compartilhar um conjunto de fatos e informações, o ensino verdadeiro é a influência de uma personalidade sobre a outra”.***

*Frank E. Gaebel*

## **AGRADECIMENTOS**

À Universidade Estadual de Campinas e à Faculdade de Odontologia de Piracicaba, na pessoa do diretor Jacks Jorge Júnior e diretor associado Alexandre Augusto Zaia e em especial ao Programa de Pós-Graduação em Materiais Dentários, pela oportunidade de desenvolver o Dinter Unioeste/UEL/Unicamp, inédito até então no Estado do Paraná.

Aos Professores Lourenço Correr Sobrinho, Mario Fernando de Goes, Simonides Consani e à coordenadora do Dinter Unioeste/UEL/Unicamp Prof<sup>a</sup>. Regina Maria Puppini Rontani, pelos momentos de aprendizado científico e pelas experiências da carreira docente trocadas, vocês sempre serão exemplo de dedicação a ser seguido.

Ao colega William Cunha Brandt por ter acompanhado todo o meu trabalho e porque sempre que precisei compartilhou seu admirável conhecimento e experiência com pesquisa.

Aos funcionários do Programa de Pós-Graduação em Materiais Dentários da FOP/UNICAMP, especialmente a Selma Aparecida Barbosa Segalla e Marcos Blanco Cangiani, muito obrigada!

As funcionárias da secretaria de Pós-Graduação da Faculdade de Odontologia de Piracicaba, em especial a Sra. Érica A. Pinho Sinhoreti, pela presteza e eficiência com que sempre nos atendeu e acolheu.

À Coordenação de Aperfeiçoamento de Pessoal de Nível superior (CAPES) e a Fundação Araucária pela contribuição financeira ao desenvolvimento do DINTER.

Aos colegas do curso de Doutorado pelas trocas de conhecimentos e pelos vários momentos agradáveis compartilhados e em especial ao colega e amigo Márcio José Mendonça que com sua perseverança e otimismo de sempre, batalhou em todos os momentos para que o projeto deste Dinter fosse possível.

Ao colegiado de Odontologia da Unioeste e especialmente aos colegas de disciplina que com companheirismo e amizade ajudaram no período em que estive afastada de sala de aula.

A todos aqueles que, embora não estejam aqui citados contribuíram para que este trabalho fosse realizado.

***Minha sincera gratidão.***

## RESUMO

O objetivo neste estudo foi analisar as propriedades mecânicas (microdureza, resistência coesiva, rugosidade e perda de massa por abrasão) e a resistência da união entre dente/restauração de diferentes resinas compostas. Para os testes de resistência da união e micro-dureza, trinta incisivos bovinos foram usados para três grupos experimentais (n=10). Cavidades tronco-cônicas (2,0 mm de altura x 2,0 mm de diâmetro maior x 1,5 mm de diâmetro menor) foram preparadas e restauradas com os seguintes sistemas adesivos e resinas compostas (Single Bond 2 e Z100; Single Bond 2 e Filtek Z350 e; P90 System Adhesive e Filtek P90 - 3M/ESPE), de acordo com as recomendações do fabricante. A fonte de luz LED Freelight 2 (3M/ESPE) foi usada para a fotoativação dos compósitos, durante 40 s. Após serem armazenados em estufa emergidos em água destilada a 37°C durante 24 h os corpos de prova receberam polimento e mensurações de microdureza (KHN) foram feitas no topo e na base ( HMV-2, Shimadzu, Tokyo). Em seguida, foram realizados nestes corpos de prova o teste de resistência de união *push-out* em uma máquina de ensaio universal (Instron modelo 4411, MA, USA). Os valores de resistência de união foram submetidos à análise de variância ANOVA 1-critério e os de KHN a análise de variância ANOVA 2-critérios, seguidos do teste de Tukey a 5%. A Filtek P90 mostrou o maior valor de resistência de união e menor em dureza Knoop diferindo estatisticamente das demais resinas compostas. A Filtek Z350 obteve valor de resistência de união e dureza Knoop intermediário entre as resinas compostas avaliadas. A Z100 obteve o menor valor de resistência de união e o maior valor de dureza Knoop diferindo estatisticamente das demais resinas compostas e diferindo entre valor de topo e base no próprio compósito. Para confeccionar as amostras para o teste de resistência coesiva (RC) foi preparado um molde de silicone com formato de ampulheta com 11 mm de comprimento, 2 mm de largura, 1 mm de espessura e 1 mm na região de constrição. Para mensuração de rugosidade de superfície (Ra) e perda de massa (PM), antes e após a escovação, foi preparado moldes de silicone com 5 mm de diâmetro e 2 mm de espessura. Todas as amostras foram polidas

com lixas de SiC 2000 e estocadas em estufa emergidas em água destilada a 37 °C por 24 h. Para Ra e PM, após o ciclo de escovação, os corpos de prova foram armazenados em estufa emergidos em água destilada a 37 °C por 24 antes da mensuração final sendo limpos em ultrassom por 15 min e removida a umidade superficial com papel absorvente. A RC foi realizada em máquina de ensaio universal (Instron modelo 4411, MA, USA). Os dados obtidos de cada teste foram submetidas a análise de variância ANOVA 1- critério e teste de Tukey a 5%. A Filtek Z350 e Z100 obtiveram os maiores valores de RC superiores a Filtek P90 e não diferindo estatisticamente entre sí. A Filtek Z350 teve o menor aumento de Ra e a Filtek P90 obteve os maiores valores de PM diferindo estatisticamente das demais resinas compostas. Pode-se concluir que tanto a matriz orgânica como a partícula de carga podem influenciar nas propriedades mecânicas e na resistência de união de restaurações de resina composta.

Palavras chave: resinas compostas, silorano, nanopartículas, propriedades mecânicas, resistência da união.

## ABSTRACT

The aim of this study was to analyze the mechanical properties (micro-hardness, cohesive strength, surface roughness and loss mass through abrasion) and the bond strength between tooth/restoration of different composite resins. For bond strength and micro-hardness analyzes, thirty bovine incisors were used for three experimental groups (n=10). Trunk- conical cavities (2,0 x 2,0 x 1,5 mm) were prepared and restored with the following adhesive systems and resin compounds (Single Bond 2 and Z100; Single Bond 2 and Filtek Z350 and; P90 System Adhesive and Filtek P90 - 3M/ESPE), following manufacture's recommendations. A LED Freelight 2 (3M/ESPE) was used for the photo-activation of the composites for 40 s. After being emerged in distilled water and stored in an incubator at 37° C for 24 hours, the specimens were polished and micro-hardness measurements (KHN) were taken at the top and base ( HMV-2, Shimadzu, Tokyo). After that, the *push-out* resistance test was performed with a universal testing machine (Instron modelo 4411, MA, USA). Bond strength values were submitted to a variance analysis ANOVA 1-way, and KHN to the variance analysis ANOVA 2-way followed by the Tukey's test at 5% level of significance. Filtek P90 showed the highest bond strength value and the lowest Knoop hardness values differing statistically from the others composite resins. Filtek Z350 obtained bond strength values and KHN values intermediate among the other resin composites. Z100 showed the lowest bond strength values and highest Knoop hardness value statically different from the others composite resins and differing from top and bottom values in itself. To prepare the specimens for the cohesive strength test (CS), an hourglass silicone mold was made measuring 11 mm in length by 2 mm wide, and 1 mm thick by 1mm wide in the narrowed region. To measure surface rugosity (Ra) and mass loss (ML) before and after brushing, silicone molds were prepared measuring 5 mm in diameter by 2 mm thick. All samples were polished with SiC 2000 sandpaper and emerged in distilled water and stored in an incubator at 37° C for 24 hours. For Ra and ML, after the brushing cycle, the specimens were emerged in distilled water and stored in an incubator at 37° C for 24 hours before

the final measurements being cleaned in ultrasonic immersion for 15 minutes and having the surface moisture removed with absorbent paper. CS was performed in a universal testing machine (Instron modelo 4411, MA, USA). The obtained data were submitted to the ANOVA 1-way analysis and the Turkey test at 5% level significance. Filtek Z350 and Z100 obtained higher CS values than Filtek P90 without statistically difference among each other. Filtek Z350 showed the lowest increase of Ra and Filtek P90 the highest ML values differing statistically from the others composite resins. It can be concluded that both the organic matrix and the type of particle filler can influence in the mechanical properties and bond strength of resin composite restorations.

Key words: composite resins, silorane, nano fillers, mechanical properties, bond strength.

## SUMÁRIO

<b>1. INTRODUÇÃO</b>	<b>1</b>
<b>2. CAPÍTULO 1:</b> <i>Bond strength and Knoop hardness of nanofilled and low shrinkage resin composites</i>	<b>4</b>
<b>CAPÍTULO 2:</b> <i>Cohesive strength, surface roughness and mass loss of composite resins with different compositions</i>	<b>18</b>
<b>3. CONSIDERAÇÕES GERAIS</b>	<b>31</b>
<b>4. CONCLUSÃO GERAL</b>	<b>33</b>
<b>REFERÊNCIAS</b>	<b>34</b>
<b>APÊNDICE</b>	<b>36</b>
<b>ANEXO</b>	<b>39</b>

## INTRODUÇÃO

O desenvolvimento das resinas compostas por Bowen, em 1962, promoveu uma evolução na Odontologia Restauradora. Atualmente as resinas compostas para restaurações conseguem mimetizar esteticamente a cor do dente natural. São relativamente estáveis no meio bucal e fáceis de manipular podendo ser fotoativadas com luz visível no espectro azul, emitida por lâmpadas halógenas ou LEDs (Jandt & Sigusch, 2009). No entanto, ainda possuem algumas desvantagens como: contração de polimerização, a qual pode levar à falha na interface resina composta/dente, acarretando muitas vezes em cáries recorrentes; maior coeficiente de expansão térmica e resistência coesiva menor do que a do substrato dental (Labella *et al.*, 1999; Brandt *et al.*, 2008; Rueggeberg, 1999).

As resinas compostas odontológicas são por definição, combinações tridimensionais de pelo menos dois materiais quimicamente diferentes, com uma interface distinta separando estes componentes (Rueggeberg, 2002; Klapdorh & Moszner, 2005; Ferracane, 2011). Uma parte é inorgânica, constituída pelas partículas de carga, cuja forma, tamanho e quantidade determinam diretamente as propriedades mecânicas das resinas compostas. Outra parte é orgânica, a matriz resinosa, basicamente constituída por monômeros resinosos que ao receberem energia na forma de luz, irão converter-se em polímeros, levando todo o conjunto do material a um estado final rígido que é a restauração propriamente dita. O silano é o agente de ligação entre a porção inorgânica e a matriz orgânica revestindo as partículas de carga e ligando-as quimicamente à matriz orgânica (Rueggeberg, 2002; Debnath *et al.*, 2004, Turssi *et al.*, 2005).

Desde o surgimento da resina composta como material restaurador de uso odontológico, várias melhorias vêm acontecendo com este material do ponto de vista de aplicabilidade clínica e longevidade destas restaurações (Rueggeberg, 2002). Basicamente dois objetivos são amplamente buscados pelos

pesquisadores no aprimoramento destes materiais: melhoria da resistência mecânica no meio bucal e longevidade do selamento das restaurações. Neste sentido, a composição deste material, tanto da matriz polimérica quanto da porção inorgânica, têm sido estudada ao longo dos anos, pois são fatores ligados diretamente ao comportamento clínico do material.

Usualmente a parte inorgânica é constituída de partículas de vidros cerâmicos, quartzo e sílica com variações no tamanho e quantidade e na matriz orgânica, monômeros de metacrilato com algumas variações de viscosidade e de grupamentos funcionais. No entanto, atualmente, duas mudanças apontam para uma nova era na composição destes materiais; uma relativa a parte inorgânica e outra na matriz orgânica (Klapdorh & Moszner, 2005; Ferracane, 2011).

Em relação à parte inorgânica, a nanotecnologia tem avançado no uso de processos químicos sintéticos para produção de partículas de carga em escala nanométrica abaixo de 100nm com o intuito de incorporar o maior conteúdo de carga inorgânica possível. Assim, é possível obter uma resina composta com melhores propriedades mecânicas que possa ser usada em região de grande esforço mastigatório e com alto polimento inicial e superior retenção desse polimento. Desse modo, como tem sido para as resinas compostas híbridas e microhíbridas, além do uso em dentes posteriores, podem ser indicadas também para as restaurações de dentes anteriores onde a exigência estética é maior (Mitra *et al.*, 2003; Ferracane, 2011).

Com relação à fase orgânica, uma nova gama de monômeros, que não apenas os derivados dos metacrilatos estão demonstrando melhorias no vedamento final das restaurações. O silorano é uma nova opção e tem conseguido menor contração de polimerização, e conseqüentemente, menor tensão de contração (Weinmann *et al.*, 2005; Palin *et al.*, 2005; Eick *et al.*, 2007; Ilie & Hickel, 2009).

Diante disso, os estudos que se propõem a comparar estas novas composições de resinas compostas com as resinas já extensivamente avaliadas na literatura e que são aplicadas clinicamente até os dias de hoje, são uma importante forma de comprovação de que estas formulações possam num futuro próximo serem empregadas rotineiramente na industrialização destes materiais, implicando em melhorias dos tratamentos restauradores.

Considerando que a avaliação *in vitro* dos materiais trazem subsídios para a posterior comparação com o desempenho clínico, a presente Tese\* está composta por dois artigos, contemplados nos capítulos 1 e 2, cujos objetivos foram, respectivamente:

1) Avaliar a resistência da união ao substrato dental e a dureza Knoop de resinas compostas com diferentes composições.

2) Avaliar a resistência coesiva, rugosidade de superfície e perda de massa das mesmas resinas compostas utilizadas no capítulo 1.

---

\* O presente trabalho está apresentado no formato alternativo de tese de acordo com as normas estabelecidas pela deliberação 002/06 da Comissão Central de Pós-Graduação da Universidade Estadual de Campinas. O artigo referente ao Capítulo 1 desta tese foi submetido ao periódico *Journal of Contemporary Dental Practice*.

## CAPÍTULO 1

### **Bond strength and Knoop hardness of nanofilled and low shrinkage resin composites.**

#### **ABSTRACT**

The aim of this study was to evaluate the Knoop Hardness (KHN) and bond strength (BS) of different resin composites. Two microhybrid (Z100 - 3M/ESPE; Filtek P90 - 3M/ESPE) and one nanofilled (Filtek Z350 - 3M/ESPE) were tested. Thirty bovine incisors were used for three experimental groups (n=10). Trunk-conical cavities were prepared in the buccal surface of each tooth with a diamond bur (#3131, KG Sorensen) at a high-speed water-cooled handpiece in a standard cavity preparation appliance (2.0 x 2.0 x 1.5 mm), resulting in a C-Factor of 2.2. Two adhesive systems were used according to manufacture instructions (Single Bond 2 and P90 System Adhesive - 3M ESPE). Restorations were made with Z100, Filtek Z350 and Filtek P90, in that order. The composites were inserted in a single increment and photoactivated with a Freelight LED unit (3M ESPE) for 40s. After photoactivation, the specimens were emerged in distilled water and stored in an incubator at 37° C for 24 hours. Microhardness measurements (KHN) were performed on the top (T) and bottom (B) of each specimen (HMV-2, Shimadzu, Tokyo). After that, the *push-out* test was performed with a universal testing machine (Instron modelo 4411, MA, USA) to evaluate bond strength. KHN mean and standard deviations were (KHN): Z100(T) - 74.1 (9.0); Z350(T) - 58.4 (3.6); P90(T)-42.8 (6,2) and Z100(B) - 66.7 (13.6); Z350(B) - 61.2 (3.6); P90(B) - 40.0 (3.0). In the KHN test data was submitted to a two way ANOVA and to Tukey's test 5%. BS mean and standard deviations were (MPa): Z100 - 12.6 (5.2), Z350 - 20.9 (6.3), P90 - 29.7 (9.0). In the BS test, the data were submitted to one way ANOVA and to Tukey's test 5%. Filtek P90 showed the highest bond strength value and the lowest Knoop hardness values differing statistically from the others composite resins. Filtek Z350 obtained bond strength values and KHN

values intermediate among the other resin composites. Z100 showed the lowest bond strength values and highest Knoop hardness value statically different from the others composite resins and differing from top and bottom values in itself.

**Keywords:** Resin composite, microhardness, fillers, organic matrix.

## INTRODUCTION

Light cured resin composites are commonly used in daily clinical practice to restore anterior and posterior teeth because of their many advantages: esthetic, bonding to tooth structure, and good mechanical properties. However, these materials undergo significant volumetric shrinkage when polymerized.<sup>1</sup> *In vitro* measurements of polymerization shrinkage of resin composites range from 1.9% to 6% and it was possible related to the resins variation composition.<sup>2</sup>

Insertion of these contracting composites into bonded preparations induces the development of mechanical stress inside the material.<sup>1</sup> The stress is transmitted via bonded interfaces to tooth structures. In light cured composites, the fast conversion induces fast increase in composite stiffness, causing high shrinkage stress at the interface.<sup>3</sup> Such stress may disrupt the bond between the composite and the cavity walls causing post-operative sensitivity or cohesive failure on the surrounding tooth tissue.<sup>4</sup>

Studies on alternative photoactivation methods have shown the beneficial effects of a modulated polymerization, and that at certain point the shrinkage stress could be controlled by the operator. However, its clinical use is difficult, because it increases the clinical time and it is dependent on the irradiance of the light curing unit, which the dentist does not usually know.<sup>1,3,6,7</sup>

Therefore, with the objective of decreasing polymerization shrinkage, and consequently, the stress generated at the tooth/restoration interface, changes in the organic matrix have been researched by industry along the years. With respect to the methacrylate resin matrix some gains have already been achieved. The most used methacrylate monomer is the BisGMA which has such a high viscosity that it must be used with a diluent monomer usually TEGDMA. Chemically, a lower molecular weight monomer such as TEGDMA, undergo a higher polymerization contraction. Thus the introduction of the BisEMA(6) and UDMA which has a high molecular weight although less C=C to be converted and higher mobility, instead of big amounts of TEGDMA, provided a reduction in the polymerization contraction.<sup>8,10</sup> Nevertheless the best approach would be the development of a monomers with reduced polymerization contraction.

Recently, a silorane-based composite (Filtek P90), a synthesized monomer originated from oxirane and siloxane, was introduced on the market. Silorane-based composites differ from the methacrylate-based composites due to the polymerization process that occurs via a cationic ring-opening reaction, which decreases the volumetric contraction of the composite when compared with other methacrylate-based composites, in which the polymerization reaction is done by addition.<sup>4,8-10</sup>

When methacrylate monomers are replaced by silorane, not only can the polymerization shrinkage be reduced, but also the stress caused by it. Thus, many problems related to composite restorations, such as microleakage and marginal staining, secondary caries and postoperative sensitivity can be overcome.<sup>9-11</sup>

Besides that, changes in the inorganic content of the methacrylate resin composites as the introduction of the nanofiller particles have already showed better results in mechanic behavior which is also an important approach to the resin composite restorative materials, not to mention the desirable controlling of the polymerization shrinkage. Therefore, the aim of this study was to evaluate the

Knoop hardness and bond strength between tooth/restoration of conventional methacrylate and silorane-based composites.

The hypotheses tested were:

a) Silorane-based composites and methacrylate-based composites would promote similar bond strength values;

b) Methacrylate-based composites will obtain higher Knoop hardness values than silorane-based composites.

## MATERIALS AND METHODS

Table 1 shows the materials.

Table 1.- Description of materials used.

<b>Resin Composite</b>	<b>Adhesive System</b>	<b>Organic Matrix</b>	<b>Filler Content (vol%)</b>	<b>Particle size (µm)</b>	<b>Manufacturer / batch</b>
Z100	Adper Single Bond 2	Bis-GMA and TEGDMA	Zirconia and silica (71%)	0.6	3M/ESPE N143694BR
Filtek Z350	Adper Single Bond 2	Bis-GMA, Bis-Ema(6), UDMA and TEGDMA	Zirconia and silica (59.5%)	nanofillers (5-20 nm) and clusters (0.6 to 1.4 µm)	3M/ESPE N178799
Filtek P90	P90 System Adhesive	Silorane (3,4-cyclohexylethyl cyclopolymethyl siloxane,bis-3,4-epoxy cyclohexylethyl phenylmethyl silane)	Quartzo and yttrium (55%)	0.47	3M/ESPE N185333

## Restorative procedures

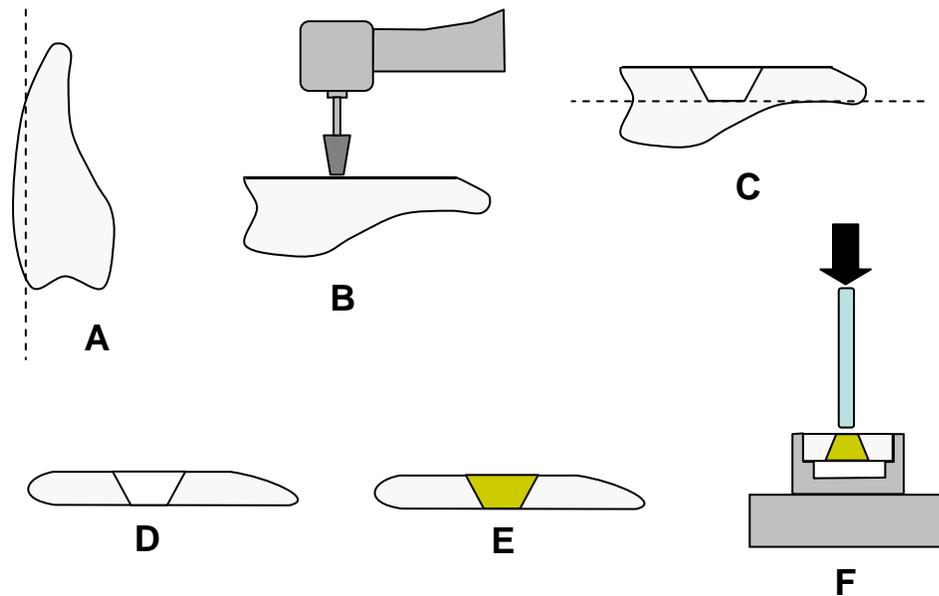
Thirty bovine incisors free from cracks or any other kind of structural defect were selected under 20x magnification. The teeth were disinfected with 0.5% chloramines for 15 days and stored for less than a month in 0.9% saline solution. The crowns were cut off at the cement-enamel junction using a double-faced diamond disk (KG Sorensen, Barueri, SP, Brazil) and the root portion were discarded. All buccal surfaces were ground and flattened (Fig. 1A) under water cooling with a 400, 600 and 1200 grit SiC paper to obtain a regular dentin surface.

Trunk-conical cavities were prepared in the buccal surface (Fig. 1B) of each tooth with a diamond bur (# 3131, KG Sorensen, Barueri, SP, Brazil) with a high-speed water-cooled hand piece in a standard cavity preparation appliance. A diamond bur was used to partially grind the lingual face of the crown and then received the same ground and flattened protocol that was done at the buccal surface (Fig. 1C). The cavity presented a tronco-conical form, measuring 2.0 mm in height, 2.0mm in diameter at the top and 1.5 mm at the bottom (Fig. 1D), resulting in a C-Factor of 2.2. The diamond bur was replaced every five preparations.

The teeth cavities were etched using 35% phosphoric acid (Scotchbond Etchant, 3M/ESPE, St. Paul, USA) and Adper Single Bond 2 adhesive system (3M/ESPE) was applied according to manufacturer's instructions and photo-activated for 10s. Ten restorations were made with Z100- 3M ESPE; Filtek Z350- 3M ESPE in A2 shade, in that order (Fig. 1E). The composite was placed in bulk mode, a polyester strip was placed over the cavity, and a microscopy acrylic slice was used to allow the composite to adapt to the preparation walls and to extrude excess material. The slice was then removed, and the light curing tip was positioned against the polyester strip, and the photo-activation was performed for 40s with a *light-emitting-diode* light source (LED) Freelight 2 (3M/ESPE) that which light irradiance was calculated using a power-meter and a digital paquimeter and was around 1000 mW/cm<sup>2</sup>.

Finally, ten restorations were made using the self-etch P90 System Adhesive, following the manufacturer's instructions, and photo-activated for 10 s.

The composite Filtek P90 (3M/ESPE) was applied with the same protocol as made with the other resins, except for the adhesive system. After photo-activated, the specimens were emerged in distilled water and stored at in an incubator at 37°C for 24 h.



**Figure 1** – Schematic representation of cavities specimens preparations. **A** – Crowns; **B** – Cavity prepared position at buccal flattened surface; **C** – Lateral view with lingual portion to be cut off; **D** – Cavity lateral view with respective dimensions: 2.0 mm in height, with a diameter of 2.0 mm at the top and 1.5 mm at the bottom; **E** – Resin composite restoration; **F** – Position to the push-out test at the universal testing machine.

### **Knoop hardness test**

After 24h, the top and bottom sample surfaces were polished under water with a 1200 grit SiC sandpaper to obtain a polished surface. Indentations

and microhardness measurements (KHN) (HNV-2, Shimadzu, Tokyo, Japan) were taken five times at each specimen surface (top and bottom) with a 50 gf load, during 15 s and the mean hardness value was obtained for each surface by the average of the five indentations.

To avoid samples dehydration and consequences on teeth/composite bond values, KHN readings were made alternately at each surface of the samples in order to keep them hydrated. First the top surface was made and the sample was putted back into the water recipe while the reading was performed at another sample. Later the bottom reading at the first sample was made one after another.

After the top and bottom readings of the three resin composites, the original mean values were submitted to a 2-way ANOVA, being the factors: material, considering the material commercial brand; and region, which analyzed the surface. The mean values, recorded as KHN, were submitted to the Tukey's test at 5% significance level.

### **Bond strength test**

Bond strength was evaluated using a push-out test. The sample was positioned on top of a metallic device containing an aperture to allow the smaller diameter of the restoration to be in contact with a cylindrical device, connected to the load cell of a universal testing machine (Instron, model 4411, Canton, MA, USA). This cylindrical device applied a compressive force on the smaller diameter surface of the restoration until rupture of the tooth–composite bond was achieved (Fig. 1F). The push-out test was carried out at a cross-head speed of 0.5 mm/min. Values were converted to MPa. The cylindrical device was positioned, so that to touch only the middle of the restoration.

After the push-out test, the original mean values were submitted to a 1-way ANOVA that analyzed material commercial brand. The mean values, recorded as MPa, were submitted to the Tukey's test at 5% significance level.

## RESULTS

Table 2 shows the KHN means comparisons. It could be noted for the Z100 composite that the top hardness was higher and statistically different from the bottom ( $p < 0.05$ ). For Filtek Z350 and P90, top and bottom did not differ ( $p > 0.05$ ). At the top region, Z100 composite showed the highest KHN means and were statistically different from the others followed by Filtek Z350 and the lowest values for Filtek P90 ( $p < 0.05$ ). At the bottom region, Filtek P90 also showed the lowest mean KHN, statistically different from Filtek Z350 and Z100 composite ( $p < 0.05$ ), which did not differ from one another ( $p > 0.05$ ).

Table 2: Hardness Knoop (KHN – kgf/mm<sup>2</sup>) means values and standard deviations of the top and bottom in the three resin composites.

Composite	Z100	Filtek Z350	Filtek P90
Region			
Top	74.1 (9.0) a,A	58.4 (3.6) a,B	42.8 (6.2) a,C
Bottom	66.7 (13.6) b,A	61.2 (3.4) a,A	40.0 (3.0) a,B

Means followed by distinct lowercase letters in column and distinct uppercase letters in line are statistically different by 5% in the level of significance according to the Tukey's test.

Table 3 shows the Values. It can be noted that Filtek P90 shows the highest bond strength mean value, differing statistically from the other composites ( $p < 0.05$ ). Z100 composite showed the lowest mean, statistically different from the other composites ( $p < 0.05$ ). Filtek Z350 had the intermediary means between the other composites.

Table 3: Bond strength (BS) mean values (MPa) and standard deviations of the three composites.

Resin Composite	Mean (sd)	Tukey (5%)
Filtek P90	29.7 (9.0)	a
Filtek Z350	20.9 (6.3)	b
Z100	12.6 (5.2)	c

Means followed by distinct letters are statistically different by 5% in the level of significance according to the Tukey's test

## DISCUSSION

Usually, the push out test is used to evaluate bond strength of endodontic cements in the radicular conduct.<sup>12,13</sup> However, in the present study, the push-out test was adapted to evaluate bond strength of restorative composites in a simulated class I cavity.

Other bond strength tests such as shear bond strength, tensile bond strength, microshear bond strength and microtensile bond strength are usually carried out to evaluate bond strength of resin composites. However, these tests are generally performed in plan surfaces. In such situation the C factor is very low and the development of the shrinkage stress is not directed to the bonding interface. The advantage of using the push out test was that the bond strength could be evaluated in a high C-factor cavity (2.2) with high stress generation directed to the bonding area.<sup>12,14</sup>

The polymerization shrinkage of dental composites is still the main cause of flaws in restorations. Shrinkage of the material can cause post-operative sensitivity and/or debonding, and consequently, marginal staining, microleakage and secondary caries.<sup>15</sup> Then, several researchers have endeavored to lessen

shrinkage stress with the objective of reducing the problems caused by polymerization shrinkage, which is inherent to the material.<sup>3,6,7</sup>

The resin composite Filtek P90 showed higher bond strength values. Filtek Z350 had intermediate values but Z100 composite obtained the lower bond strength values probably due to the high filler content and the resin matrix composition with TEGDMA and BisGMA which imparts toughness that might increase the tensile imparted to the tooth/restoration interface. The substitution in part of the BisGMA with BisEMA(6) in Filtek Z350 composition decreased the resin viscosity thus a small amount of TEGDMA was necessary so decreasing the polymerization shrinkage. Also the incorporation of UDMA which is a high molecular weight monomer enabled a lower polymerization shrinkage and contraction stress which favored a better bond strength result.

The increase in bond strength values are directly related to a lower shrinkage contraction. The very distinct behavior of the Silorane network is generated by the cationic ring opening polymerization of the cycloaliphatic oxirane moieties, which stand for their low shrinkage and low polymerization stress. The oxirane monomer rings expand when opened thus minimizing the contraction process that happens when the monomers get near each other. With the methacrylate resins, the decrease of the distance between the monomer molecules during the polymerization process, undergo a shrinkage phenomenon.<sup>8-10,19</sup> Consequently, the first hypothesis was rejected.

The second hypothesis was accepted since methacrylate-based composites obtained higher KNH than the silorane-based composites. Knoop hardness is directly related to the inorganic filler content in volume which could explain the higher values for Z100 in the top surface differing statistically from the others. However the higher filler amount in Z100 must have inhibited internal light absorption leading to a different KHN between top and bottom surfaces which did not happened with Filtek Z350 that showed the intermediate top surface value among the others but without difference to the bottom. Filtek P90 presented the lowest top surface KNH values in relation to the others composites nevertheless

also without statistically difference to the bottom surface. Those results also indicated that the monomers composition and not only the filler content can influence changes in the KHN between top and bottom surfaces.

On the other hand, the lower Filtek P90 KHN's can suggest a composite with inferior mechanical properties. It can be related to a possible lower degree of conversion. Also further studies comparing the degree of conversion with mechanical properties such as wear and ultimate tensile strength, should be conducted with this resin composite. The ideal to a low shrinkage composite is that it should be accompanied by either lower stress shrinkage and higher bond strength numbers and optimal mechanical properties.

Finally, it is important to point out some limitations of this study. The use of bovine teeth implies in caution on the interpretation of the results. However, the objective of this study was to evaluate the behavior of the different composition composites under confinement phase. In addition, the use of bovine incisors is supported by several authors.<sup>16-18</sup>

Filtek P90 has its own adhesive system, because it possesses a different composition to the methacrylate-based composites like Filtek Z350 and Z100.<sup>20-22</sup> P90 system adhesive is a self-etch adhesive differently from Single Bond 2 used with Filtek Z350 and Z100 that is an etch-and-rinse adhesive. The use of different adhesive systems might have contributed to the differences found in the bond strength values. It could be suggested to further studies also a comparison among the P90 system adhesive and others self-etch adhesive systems.

## **CONCLUSION**

Differences in the composition of the composite resins influenced in the Knoop hardness and bond strength of restorations.

The composite resin Z100 showed the highest KHN values, but the lowest bond strength values; while the Filtek P90 showed the lowest KHN values, but the highest bond strength values. The Filtek Z350 obtained intermediate values for both KHN and bond strength.

## REFERENCES

- 1- Davidson CL & de Gee AJ (1984) Relaxation of polymerization contraction stresses by flow in dental composites *Journal of Dental Research* 63 146-148.
- 2- Labella R, Lambrechts P, Van Meerbeek B & Vanherle G (1999) Polymerization shrinkage and elasticity of flowable composites and filled adhesives *Dental Materials* 15 128-137.
- 3- Brandt WC, de Moraes RR, Correr-Sobrinho L, Sinhoreti MA & Consani S (2008) Effect of different photo-activation methods on push out force, hardness and cross-link density of resin composite restorations *Dental Materials* 24 846-850.
- 4- Tezvergil-Mutluay A, Lassila LV & Vallittu PK (2008) Incremental layers bonding of silorane composite: the initial bonding properties *Journal of Dentistry* 36 560-3
- 5- Emami N, Soderholm KJ & Berglund LA (2003) Effect of light power density variations on bulk curing properties of dental composites *Journal of Dentistry* 31 189-196.
- 6- Segreto D, Brandt WC, Correr-Sobrinho L, Sinhoreti MA & Consani S (2008) Influence of irradiance on the push-out bond strength of composite restorations photoactivated by LED *Journal of Contemporary Dental Practice* 9 89-96.
- 7- Alonso RC, Cunha LG, Correr GM, Cunha Brandt W, Correr-Sobrinho L & Sinhoreti MA (2006) Relationship between bond strength and marginal and internal adaptation of composite restorations photocured by different methods *Acta Odontologica Scandinavica* 64 306-313.

- 8- Palin WM, Fleming GJ, Nathwani H, Burke FJ & Randall RC (2005) In vitro cuspal deflection and microleakage of maxillary premolars restored with novel low-shrink dental composites *Dental Materials* 21 324-335.
- 9- Weinmann W, Thalacker C & Guggenberger R (2005) Siloranes in dental composites *Dental Material* 21 68-74.
- 10- Eick JD, Kotha SP, Chappelow CC, Kilway KV, Giese GJ, Glaros AG & Pinzino CS (2007) Properties of silorane-based dental resins and composites containing a stress-reducing monomer *Dental Materials* 23 1011-1017.
- 11- Ernst CP, Meyer GR, Klocker K & Willershausen B (2004) Determination of polymerization shrinkage stress by means of a photoelastic investigation *Dental Materials* 20 313-321.
- 12- Perdigao J, Geraldeli S & Lee IK (2004) Push-out bond strengths of tooth-colored posts bonded with different adhesive systems *American Journal of Dentistry* 17 422-426.
- 13- Kurtz JS, Perdigao J, Geraldeli S, Hodges JS & Bowles WR (2003) Bond strengths of tooth-colored posts, effect of sealer, dentin adhesive, and root region *American Journal of Dentistry* 16 31A-36A.
- 14- Feilzer AJ, de Gee AJ & Davidson CL (1990) Quantitative determination of stress reduction by flow in composite restorations *Dental Materials* 6 167-171.
- 15- Cunha LG, Alonso RC, Neves AC, de Goes MF, Ferracane JL & Sinhoreti MA (2009) Degree of conversion and contraction stress development of a resin composite irradiated using halogen and LED at two C-factor levels *Operative Dentistry* 34 24-31.
- 16- Nakamichi I, Iwaru M & Fusayama T (1983) Bovine teeth as possible substitutes in the adhesion test *Journal of Dental Research* 62 1076-81.
- 17- Reeves GW, Fitchie JG, Hembree JH Jr & Puckett AD (1995) Microleakage of new dentin bonding systems using human and bovine teeth *Operative Dentistry* 20 230-235.

- 18- Schilke R, Lisson JA, Bauss O & Geurtsen W (2000) Comparison of the number and diameter of dentinal tubules in human and bovine dentine by scanning electron microscopic investigation *Archives of Oral Biology* 45 355-361.
- 19- Ilie N, Hickel R (2011) Resin composite restorative materials *Aust Dent J* 56 Suppl 1 59-66.
- 20- Guiraldo RD, Consani S, Consani RL, Berger SB, Mendes WB, Sinhoreti MA, Correr-Sobrinho L (2010) Comparison of silorane and methacrylate-based composite resins on the curing light transmission *Braz Dent J* 21 538-42
- 21- Boaro LC, Gonçalves F, Guimarães TC, Ferracane JL, Versluis A, Braga RR (2010) Polymerization stress, shrinkage and elastic modulus of current low-shrinkage restorative composites *Dent Mat* 26 1144-50
- 22- Al- Boni R, Raja Ola M (2011) Microleakage evaluation of silorane based composite versus methacrylate based composite *Journal of Conservative Dentistry* 13 152-155

## CAPÍTULO 2

### **Cohesive strength, surface roughness and mass loss of composite resins with different compositions.**

#### ABSTRACT

The aim on this study was to evaluate the cohesive strength (CS), surface roughness (Ra) and mass loss (ML) of three composite resins; Filtek Z350 (3M/ESPE), Filtek P90 (3M/ESPE) and Z100 (3M/ESPE). To prepare the specimens for the CS test, an hourglass silicone mold measuring 11 mm by length, 2 mm wide, and 1 mm thick by 1mm wide in the narrowed region was constructed and the test was performed at an universal testing machine (Instron, model 4411, Canton, MA, USA). Also, ten specimens for each resin composite were prepared to do the Ra test and ML evaluation. It was obtained through a silicone mode with spherical shape constructed with 5 mm in diameter and 2 mm in thickness. The photo-activation was performed for 40 s with a LED unit, Freelight 2 (3M/ESPE), with an irradiance of 1000 mW/cm<sup>2</sup>. After the photo-activation, the specimens were removed and a slightly finishing was applied with 2000 SiC sandpaper. Then, the specimens were cleaned in the ultrasonic immersion for 15 minutes. All the specimens were emerged in distilled water and stored in an incubator at 37° C for 24 hours. After the storage period, the specimens were dried with paper pallets the initial mass weight was registered with an analytic precision balance and then the initial surface roughness reading was made. Later, the specimens were submitted to brushing at 30,000 cycles for specimen and cleaned with ultrasonic immersion for 15 minutes and emerged in distilled water and stored in an incubator at 37° C for 24 hours. After the final storage period the specimens were again dried with absorbent paper pallets to remove the water adsorbed at the surface. The final mass weight was registered and the last surface roughness reading was performed. The mean values obtained for each test were as follows: CS (MPa)- Z100=50.8; Z350=52.0; P90=39.3; Ra(μm) Z100=0.3257; Z350=0.0469; P90=0.2262; ML (mg)- Z100=0.00041; Z350=0.00059; P90=0.00110. The data were submitted to 1-way ANOVA and to Tukey's test. Z100 and Filtek Z350 showed the highest CS means and did not differ from each other. Filtek P90 showed the lowest mean being statistically different from the others. Z100 obtained the highest Ra mean value, differing statistically from the

others; Filtek Z350 the lowest mean and Filtek P90 an intermediary mean. Z100 and Filtek Z350 were similar statistically with the lowest ML means and Filtek P90 had the highest ML mean being different statistically from the others. It was concluded that differences in the composition of the organic matrix or in the particle fillers change the mechanical properties of composite resins.

**Keywords:** resin composite, mass loss, surface roughness, cohesive strength.

## INTRODUCTION

Restorative resin composites have been used in dentistry for more than 40 years. In spite of the undeniable technological advances introduced during the last decades, mechanical and bond strengths remain one of the most studied topics for clinical performance.<sup>1,2</sup>

By definition, a composite is a material that consists of two or more components. Typically, dental resin filling composites contain 15-25% vol. of a free-radically polymerizable organic matrix and 30-70% vol. of a mixture of different inorganic fillers, in addition to a photo-initiator system or, in some cases, other curing systems and further additives such as stabilizers and pigments.<sup>1,2</sup>

The resin composites are similar in that they are all composed of a polymeric matrix, typically dimethacrylate, reinforcing fillers, typically made from radiopaque glass, a silane coupling agent for bonding the filler to the matrix, and chemicals that promote or modulate the polymerization reaction.<sup>1,2,17</sup>

Mechanic strengths are directly dependent on the inorganic filler content. Used fillers are characterized by different manufacturing techniques, the average particle size and the chemical composition.<sup>2,3</sup> The newly nanotechnology applied to inorganic filler development it has been showing improvements in the mechanical and optical properties of the dental materials.<sup>3,9,10</sup> A better maintenance of polish with nano filled composites, when compared to the conventional composite resins, have been reported.<sup>13,14,16</sup>

However the polymerization shrinkage and the stress generated with it is still considered as being their main drawback.<sup>5,6</sup> The clinical consequences as imperfections in marginal adaptation and the appearance of recurrent caries, constitute the main reasons for premature replacement of resin composite restorations. This explains why it is regarded as the main limitation of present-day resin composites.<sup>7</sup>

During polymerization of the methacrylate resins, the viscous liquid gradually transforms into a rigid material by radical polymerization involving the double bonds C=C of methacrylate groups. This polymerization involves a volume shrinkage which has mainly originated by a chemical contraction which is attributed to a change in inter-atomic spacing between monomer molecules. Furthermore, the extension of polymerization shrinkage depends, among other things on the relative mobility, the molecular weight and functionality of the monomers involved. As the higher the concentration of high molecular weight monomers, the lower the amount of carbon double bonds per unit volume. In addition, high molecular weight monomers in generally present lower mobility, which reduces the final degree of conversion reached by the composite, also contributing to a lower shrinkage.<sup>7,17</sup>

In 2007, a silorane-based composite became commercially available. The silorane molecule presents a siloxane core with four oxirane rings attached to it that open upon polymerization to bond to other monomers. The oxirane ring opening causes a volumetric expansion that partially compensates the shrinkage resultant from molecular bonding.<sup>8,17,19</sup> Literature data confirmed that a silorane-based commercial composite presents less than 1.0% of total volumetric shrinkage, compared to 2.0–3.5% for BisGMA based composites, causing less tooth deflection and microleakage. Sometimes its mechanical properties are comparable to those of dimethacrylate-based materials.<sup>5,8</sup>

However, the main target in the development of the silorane-based composite is to decrease shrinkage stress, this should not be the detrimental factor of the mechanical properties. Since low shrinkage and high mechanical properties are generally opposite factors, this study aimed to analyze mechanical behavior.

In view of the above explanation, this study was to make a comparative analysis between conventional composite resins with those with newer compositions as it is important to compare the newer composites with those showing long laboratory and clinical track records and of course, more physical data have to be taken into consideration for the complete evaluation of the overall performance of a dental restorative composite.

In that way, the objective of that study was to compare the cohesive strength, surface roughness and mass loss among three different composite resins, when different technologies as organic matrix based on silorane and particles fillers in the nanometric scale were included. The null hypothesis should be that results for the new resin composite compositions are not different from regular conventional composites.

## MATERIALS AND METHODS

The materials used are shown in Table 1.

Table 1. Materials used.

<b>Resin Composite</b>	<b>Adhesive System</b>	<b>Organic Matrix</b>	<b>Filler Content (vol%)</b>	<b>Particle size (µm)</b>	<b>Manufacturer / batch</b>
Z100	Adper Single Bond 2	Bis-GMA and TEGDMA	Zirconia and silica (71%)	0.6	3M/ESPE N143694BR
Filtek Z350	Adper Single Bond 2	Bis-GMA, Bis-Ema(6), UDMA and TEGDMA	Zirconia and silica (59.5%)	nanofillers (5-20 nm) and clusters (0.6 to 1.4 µm)	3M/ESPE N178799
Filtek P90	P90 System Adhesive	Silorane (3,4-epoxy cyclohexylethyl cyclopolymethyl siloxane,bis-3,4-epoxy cyclohexylethyl phenylmethyl silane)	Quartzo and yttrium (55%)	0.47	3M/ESPE N185333

### **Cohesive Strength Test**

In order to obtain the specimens for the cohesive strength test, an hourglass silicone mold was constructed measuring 11 mm in length by 2 mm wide, and 1 mm thick by with 1mm wide in the constricted region. The resin composite was adapted directly inside the silicone mold with a spatula until the complete filling of the mold. A polyester strip was placed on top of the resin surface so as to achieve uniform thickness of the material and to allow direct contact with the light source. Light activation was performed for 40 s with a LED unit, FreeLight 2 (3M/ESPE), with an irradiance of 1000 mW/cm<sup>2</sup>. Ten specimens were prepared with each resin composite. After light curing, the specimens were carefully removed from the silicone mold and a slight finishing was made with 2000 SiC sandpaper to remove some resin composite excess. All specimens were stored at 37°C in distilled water for 24h. The cohesive strength test was performed with a universal testing machine (Instron, model 4411, Canton, MA, USA). The test was conducted at a cross-head speed of 0.5 mm/min with a 500N loading cell, up to the moment of fracture. Thus, the registered values (in kgf) were converted to MPa. The datas were submitted to a 1-way ANOVA. The mean values were submitted to the Tukey test at a 5%-significance level.

### **Surface Roughness (Ra) Test and Mass Loss (ML) Evaluation**

The specimens for mass loss evaluation and surface roughness test were the same. They were obtained by building a silicone mold measuring 5 mm in diameter and 2 mm in thickness. The resin was inserted into the mold in a single increment. A polyester strip was placed on top of the resin surface so as to achieve uniform thickness of the material and to allow direct contact with the light source. Light activation was performed for 40s with a LED unit, FreeLight 2 (3M/ESPE), with an irradiance of 1000 mW/cm<sup>2</sup>. Ten specimens were prepared from each resin composite. After the light curing, the specimens were carefully removed from the silicone mold and a slight finishing was made with polishing discs to remove some lateral resin composite excess. Then, the specimens were cleaned in ultrasonic

immersion for 15 minutes. The bottom surface was marked and all the specimens were stored at 37°C in distilled water for 24 h.

After the storage period, the specimens were dried with absorption paper pallets and the initial mass weight for each specimen was registered with an analytic precision scale Chyo model JK-180. Soon after, the initial surface roughness test was made with a rugosimeter Surfcoorder SE1700 (Kosaka Corp., Tokyo, Japan) with a 2 µm diamond stylus employing a cut-off length of 0.25 mm, a measuring length of 2 mm, at a speed of 0.5 mm/s. Preliminary testing was performed to evaluate the specimens for defects (i.e., cracks, air bubbles) under a stereomicroscope at a magnification of 100X. The surfaces that were free from defects were tested by taking a reading at the center of each specimen. Three recordings were made per specimen surface. The roughness parameter was evaluated as the arithmetic mean of the sum of the roughness profile values (Ra). The roughness means were recorded as the representative data value for each specimen.

Soon after, the specimens were subjected to brushing with Colgate® brand toothbrush and Sorriso® brand toothpaste, using a brushing machine (Equilabor, Piracicaba, SP, Brazil) set at a load of 200 gf at a frequency of 250 strokes/min for 30,000 strokes. The specimen was placed into a silicone holder which in turn was placed into the metal frame of the brushing machine. The specimen was brushed in a linear motion in a chamber containing a mixture of 6 g of toothpaste and 6 ml of distilled water. After that, the specimens were cleaned in ultrasonic immersion for 15 minutes and again stored at 37°C in distilled water for 24 h.

After storage period, the specimens were again dried with absorbent paper pallet to remove the water adsorbed at the surface. The second mass weight was registered for each specimen and then, the final surface roughness reading was performed as well as the initial.

Concerning the Ra test, the original data after the brushing cycle were submitted to a 1-way ANOVA. The mean values, recorded in µm as Ra values,

were submitted to the Tukey test at a 5%-significance level. As well to the mass loss evaluation (mg) after the brushing cycle, the original data were submitted to a 1-way ANOVA and the recorded mean values were submitted to the Tukey test at a 5%-significance level.

## RESULTS

Table 2 shows the cohesive strength mean comparisons. It can be seen that resin composites Z100 and Filtek Z350 showed the highest CS means and did not differ from each other ( $p>0.05$ ). The Filtek P90 showed the lowest mean being statistically different from the others ( $p<0.05$ ).

Table 2: Cohesive strength means (MPa) and standard deviation for the three resin composites.

Composite	Mean (sd)	Tukey (5%)
Filtek Z350	52.0 (5.3)	A
Z100	50.8 (11.1)	A
Filtek P90	39.3 (11.6)	B

Means followed by distinct letters are statistically different in 5% level significance according to the Tukey's test.

Table 3 shows the mean comparisons of increasing surface roughness (Ra). It can be noted that Z100 composite showed the highest Ra mean value, differing statistically from the other composites ( $p<0.05$ ). Filtek Z350 showed the lowest mean differing statistically from the other composites ( $p<0.05$ ). Filtek P90 obtained an intermediary mean compared to the other composites.

Table 3: Surface roughness increasing means ( $\mu\text{m}$ ) and standard deviations for the three composites.

Composite	Mean (sd)	Tukey (5%)
Z100	0.3257 (0.1426)	A
Filtek P90	0.2262 (0.0837)	B
Filtek Z350	0.0469 (0.0038)	C

Means followed by distinct letters are statistically different in 5% level significance according to the Tukey's test.

Table 4 shows the means mass loss. It can be observed that the resin composite Z100 and Filtek Z350 showed the lowest mass loss means and did not differ statistically from each other ( $p>0.05$ ). Filtek P90 showed the highest mass loss mean being different statistically from the others ( $p<0.05$ ).

Table 4: Mass loss means (mg) and standard deviation for the three resin composites.

Resin composite	Mean (sd)	Tukey (5%)
Filtek P90	0.00110 (0.00014)	A
Filtek Z350	0.00059 (0.00012)	B
Z100	0.00041 (0.00032)	B

Means followed by distinct letters are statistically different at a 5% level of significance according to the Tukey's test.

## DISCUSSION

In the present study, in vitro evaluation of three different resin composites, allowed to relate the results obtained directly with the composition

present in them; size of particles, amount of filler and type of organic matrix can influence in the results.<sup>9,10</sup> In this manner, the intrinsic behavior of each material can be related with the analysis of the two constituent parts of the composites; organic matrix and inorganic filler.<sup>11</sup>

Many studies have demonstrated the relation of the filler content in the mechanical properties of the composite resins.<sup>2,3,5</sup> The primary objective of filler presence is to give strength and reduce the amount of organic matrix. The volume content of inorganic fillers are directly related to important resin properties such as: increase of hardness and strength, wearing decrease; decrease of polymerization contraction; decrease of thermal contraction and expansion; increase in viscosity for better handling of the material; decrease of water sorption, softening and staining; increase of radiopacity.<sup>2</sup>

Initially, in the cohesive strength test, the exercised force is traction in the long axis of the specimen, that tends to stretch out or to prolong the body until the moment of the fracture. Therefore, the evaluation of the cohesive strength is constituted in primordial factor to correlate with the quality of the formed polymer<sup>12</sup>. In this test, the composite resins Filtek Z350 and Z100 showed the largest averages of CS and they didn't differ amongst themselves although they have differences in filler volume content and in the organic matrix composition.

In spite of Z100 resin composite having a higher inorganic content in volume (Z100=71%; Filtek Z350=59.5%), Filtek Z350 has an average particle size in the nano scale from 5 to 20 nm. This observation is made because the smaller the particle filler, the bigger the amount of inorganic filler that can be incorporated at the resin composite, and thus, the mechanical properties are improved.

Considering the organic matrix, Z100 has a large amount of TEGDMA which can impart toughness to the organic matrix. The Filtek Z350 had the TEGDMA substituted largely by the Bis-EMA(6) and has the presence of UDMA. These monomers have a high molecular weight but are more flexible molecule due to the absent of hydroxyl (OH) group thus collaborating to the polymerization

propagation. This may explain the statistically similar CS behavior among Z100 and Filtek Z350.

The resin composite Filtek P90 presented the lowest CS values among the other resin composites. If one compares the filler particle geometry, the Filtek P90 resin composite, made of quartz and yttrium fluoride with irregular geometry, could have the filler working as an inductor in the tension concentration more easily, helping to provoke a premature fracture in the composite thus reducing the CS of the resin composite. The Filtek Z350 and Z100 have different filler particle geometry from both zirconia and silica which are a more favorable round geometry.

Also, quite different in the silorane resin composite is the silanization fillers particles technology. The efficiency of this silane/fillers bond might have influenced in the CS values as well as in the ML values to the Filtek P90. A weak chemical bond in the silane/filler interface may lead to a more easily loss of the inorganic filler and consequently mass loss. Thus, more comparative evaluations of the silanization effectiveness in the silorane and metacrylate resins are required.

Furthermore, the distinct cationic ring opening polymerization chemistry in silorane, comparing to the metacrylate resins, may have a different speed in polymerization propagation that can influence in the final quality of the formed polymer. A further evaluation of the degree of conversion related to the CS and ML could be indicated.

Considering the surface roughness values obtained in this study, it was observed that, after the brushing cycle, the largest increase of surface roughness happened for Z100. In this aspect, particle size has a plenty influence, because the bigger the particle size, the bigger is the flaw left in the resin when the particle stands out of it. The resin composite Filtek Z350 presented the lowest increasing Ra surface differing from the others and corroborating with the study of Costa et al.,<sup>13</sup> that obtained the largest Ra with the resin Z100 and the smallest values for the Filtek Supreme composite, that presents identical composition to Filtek Z350; this is due to the nanotechnology which provides a bigger filler volume content with size in the nano scale so the wear resistance is higher and so is the finishing

maintenance. The composite Filtek P90 obtained intermediate Ra average in relation to the other composites.

It was reported that there are no differences between Ra of nanohybrid and microhybrid composites when compared with the nanofillers<sup>14</sup>. However the study of Senawongse and Pongprueksa<sup>15</sup> showed that Filtek Z350 did not obtain increase of surface roughness after brushing in relation to other microhybrid and nanohybrid composites. The reason for that was because the nanofillers composites have an average of particles size smaller than the ones present in the microhybrid and nanohybrid resin composite here evaluated.<sup>3,18,19</sup> Correlation between filler size and surface roughness was done in the present study as well in others.<sup>16</sup>

Then, in agreement with the obtained results, the hypothesis that there would not be differences among the composition of silorane based resin composites and the traditionally based in methacrylate was rejected.

## **CONCLUSION**

Differences in the composition of the organic matrix or in the particle fillers change the mechanical properties of composite resins.

The composite resin nanofilled Filtek Z350 showed the highest cohesive strength and the lowest mass loss and increasing of surface roughness.

## **REFERENCES**

- 1- Ferracane JL. Resin composite--state of the art. Dent Mater. 2011;27(1):29-38.
- 2- Klapdorh S, Moszner N. New inorganic components for dental filling composites. Monatsh Chem 2005; 136:21-45.
- 3- Mitra SB, Wu D, Holmes BN. An application of nanotechnology in advanced dental materials. J Am Dent Assoc. 2003;134(10):1382-90.

- 4- Brandt WC, de Moraes RR, Correr-Sobrinho L, Sinhoreti MA, Consani S. Effect of different photo-activation methods on push out force, hardness and cross-link density of resin composite restorations. *Dent Mater.* 2008 Jun;24(6):846-50.
- 5- Ilie N, Hickel R. Macro-, micro- and nano-mechanical investigations on silorane and methacrylate-based composites. *Dent Mater.* 2009;25(6):810-9.
- 6- Ilie N, Hickel R. Silorane-based dental composite: behavior and abilities. *Dent Mater J.* 2006;25(3):445-54.
- 7- Dewaele M, Truffier-Boutry D, Devaux J, Leloup G. Volume contraction in photocured dental resins: the shrinkage-conversion relationship revisited. *Dent Mater.* 2006;22(4):359-65.
- 8- Weinmann W, Thalacker C, Guggenberger R. Siloranes in dental composites. *Dent Mater.* 2005;21(1):68-74.
- 9- Lu H, Roeder LB, Powers JM. Effect of polishing systems on the surface roughness of microhybrid composites. *J Esthet Restor Dent.* 2003;15(5):297-303
- 10- Scheibe KG, Almeida KG, Medeiros IS, Costa JF, Alves CM. Effect of different polishing systems on the surface roughness of microhybrid composites. *J Appl Oral Sci.* 2009;17(1):21-6.
- 11- Lutz F, Phillips RW. A classification and evaluation of composite resin systems. *J Prosthet Dent.* 1983;50(4):480-8.
- 12- Miyazaki M, Oshida Y, Moore BK, Onose H. Effect of light exposure on fracture toughness and flexural strength of light-cured composites. *Dent Mater.* 1996;12(6):328-32.
- 13- Da Costa J, Ferracane J, Paravina RD, Mazur RF, Roeder L. The effect of different polishing systems on surface roughness and gloss of various resin composites. *J Esthet Restor Dent.* 2007;19(4):214-24.
- 14- da Silva JM, da Rocha DM, Travassos AC, Fernandes VV Jr, Rodrigues JR. Effect of different finishing times on surface roughness and maintenance of

- polish in nanoparticle and microhybrid composite resins. *Eur J Esthet Dent.* 2010;5(3):288-98.
- 15- Senawongse P, Pongprueksa P. Surface roughness of nanofill and nanohybrid resin composites after polishing and brushing. *J Esthet Restor Dent.* 2007;19(5):265-73.
- 16- Tjan AH, Chan CA. The polishability of posterior composites. *J Prosthet Dent.* 1989;61(2):138-46.
- 17- Ilie N, Hickel R (2011) Resin composite restorative materials *Aust Dent J* 56 Suppl 1 59-66
- 18- Aguiar FH, Georgetto MH, Soares GP, Catelan A, Dos Santos PH, Ambrosano GM, Figueroba SR, Lovadino JR (2011) Effect of different light-curing modes on degree of conversion, staining susceptibility and stain's retention using different beverages in a nanofilled composite resin *J Esthet Restor Dent* 23 106-14
- 19- Guiraldo RD, Consani S, Consani RL, Berger SB, Mendes WB, Sinhoreti MA, Correr-Sobrinho L (2010) Comparison of silorane and methacrylate-based composite resins on the curing light transmission *Braz Dent J* 21 538-42

### 3. CONSIDERAÇÕES GERAIS

Sempre que novos materiais são lançados no mercado, inúmeras dúvidas com relação ao uso também são “lançadas” entre os cirurgiões-dentistas. Além disso, com a possibilidade de inovação de técnicas devido à solução de problemas tradicionais dentro da clínica odontológica, muitas são as expectativas com relação a esses novos materiais.

Na confecção de restaurações de resina composta, a existência da contração de polimerização deve ser considerada. Esta contração pode causar falha nas restaurações como por exemplo, a desunião entre o dente e a restauração, o aparecimento de cáries recorrentes, o manchamento das margens das restaurações, sensibilidade pós-operatória, e conseqüentemente, a substituição da restauração.

Com o intuito de eliminar ou pelo menos diminuir a problemática da contração de polimerização, um novo monômero chamado silorano foi incorporado à matriz orgânica da resina composta, a Filtek P90. De acordo com esse trabalho, pode-se verificar que a proposta de diminuição da contração de polimerização com o uso do silorano na Filtek P90 e, conseqüente melhora da união com o substrato dental, mostrou-se eficaz. Pois foi a resina composta que obteve os maiores valores de resistência da união entre o dente e a restauração. Mas na avaliação de outras propriedades como dureza Knoop, resistência coesiva, rugosidade e perda de massa, Filtek P90 não mostrou valores que a colocassem como superior às outras resinas compostas Z100 e Filtek Z350.

Além da longevidade das restaurações, todo profissional busca uma excelência estética em seus trabalhos clínicos. Isso se deve não somente à busca da satisfação profissional, mas também devido à crescente demanda estética dos pacientes atuais. Dessa forma, um produto que permitisse alto nível de polimento e principalmente a manutenção desse polimento é desejável. Assim, a incorporação de partículas de carga na escala nanométrica foi realizada na resina composta Filtek Z350. Um dos objetivos da incorporação de nanopartículas era

fazer com que durante o desgaste inerente ao uso da restauração no ambiente bucal, ocorresse um mínimo de imperfeições. De acordo com este estudo, o propósito foi alcançado, pois Filtek Z350 mostrou os menores valores de rugosidade após desgaste por escovação com resultados satisfatórios nas outras propriedades, com exceção dos valores de resistência da união que foram inferiores ao da Filtek P90. Dessa forma, pode-se sugerir a utilização da resina composta Filtek Z350 para restaurações anteriores, pois além de propriedades mecânicas adequadas, possui excelente polimento e manutenção desse polimento. Sua indicação para dentes posteriores também é pertinente, pois possui boas propriedades mecânicas para esta região; além do que, apesar da manutenção de polimento não ser uma propriedade de “extrema importância” do ponto de vista estético nessa região, auxilia em uma menor retenção de biofilme bacteriano sobre a restauração.

Com relação à resina composta Filtek Z100 pode-se observar que sua utilização e/ou indicação quando comparada as resinas compostas Filtek Z350 e Filtek P90 deve ser cuidadosa no sentido de tentar minimizar a contração de polimerização e a conseqüente tensão de contração transmitida à interface dente/restauração por parte do cirurgião dentista respeitando-se o fator C de configuração cavitária, o uso de técnica incremental e de métodos de fotoativação que permitam minimizar a contração de polimerização bastante importante deste material em relação aos demais avaliados neste estudo que resultaram nos menores valores de resistência da união.

A Z100 não mostrou propriedades superiores à Filtek Z350, com exceção à dureza Knoop. Este dado aponta para uma tendência a fabricação de resinas com monômeros de maior peso molecular porém com viscosidade que dispense a necessidade de diluentes como o TEGDMA presente em grande quantidade na resina Z100 e que parece estar diretamente relacionado a maior contração de polimerização e maior tensão nesta resina composta.

#### 4. CONCLUSÃO GERAL

Dentro das limitações desse estudo, e diante dos resultados obtidos foi possível concluir que:

- A Filtek P90 mostrou maiores resultados de resistência de união, menores valores de dureza Knoop, resistência coesiva e valor intermediário de aumento de rugosidade de superfície em relação a resina Z100 e a Filtek Z350. A perda de massa da Filtek P90 foi superior em relação às demais resinas compostas avaliadas.

- A Filtek Z350 mostrou melhores resultados de rugosidade após desgaste por escovação e perda de massa que as outras resinas compostas avaliadas. No entanto, obteve valores de resistência da união inferiores à Filtek P90.

- A resina composta Z100, com exceção dos valores de dureza Knoop, não mostrou destaque em nenhuma das avaliações desse estudo.

## REFERENCIAS\*

Bowen RL. Dental filling material comprising vinyl silane treated fused silica and a binder consisting of the reaction product of bisphenol and glycidyl acrylate. US n. 3066112.27 1962:

Brandt WC, de Moraes RR, Correr-Sobrinho L, Sinhoreti MA, Consani S. Effect of different photo-activation methods on push out force, hardness and cross-link density of resin composite restorations. *Dent Mater.* 2008 Jun;24(6):846-50.

Debnath S, Ranade R, Wunder SL, McCool J, Boberick K, Baran G. Interface effects on mechanical properties of particle-reinforced composites. *Dent Mater.* 2004;20(7):677-86.

Eick JD, Kotha SP, Chappelow CC, Kilway KV, Giese GJ, Glaros AG & Pinzino CS. Properties of silorane-based dental resins and composites containing a stress-reducing monomer. *Dent Mater.* 2007;23:1011-1017.

Ferracane JL. Resin composite--state of the art. *Dent Mater.* 2011;27(1):29-38.

Ilie N, Hickel R. Macro-, micro- and nano-mechanical investigations on silorane and methacrylate-based composites. *Dent Mater.* 2009;25(6):810-9.

Jandt KD, Sigusch BW. Future perspectives of resin-based dental materials. *Dent Mater.* 2009;25(8):1001-6.

---

\* De acordo com a norma da UNICAMP/FOP, baseada na norma do International Committee of Medical Journal Editors – Grupo de Vancouver. Abreviatura dos periódicos em conformidade com o Medline.

Klapdorh S, Moszner N. New inorganic components for dental filling composites. *Monatsh Chem* 2005; 136:21-45.

Labella R, Lambrechts P, Van Meerbeek B & Vanherle G. Polymerization shrinkage and elasticity of flowable composites and filled adhesives. *Dent Mater.* 1999;15:128-137.

Mitra SB, Wu D, Holmes BN. An application of nanotechnology in advanced dental materials. *J Am Dent Assoc.* 2003;134(10):1382-90.

Palin WM, Fleming GJ, Nathwani H, Burke FJ & Randall RC. In vitro cuspal deflection and microleakage of maxillary premolars restored with novel low-shrink dental composites. *Dent Mater.* 2005;21:324-335.

Rueggeberg F. Contemporary issues in photocuring. *Compend Contin Educ Dent Suppl* 1999; (25): S4-15; quiz S73.

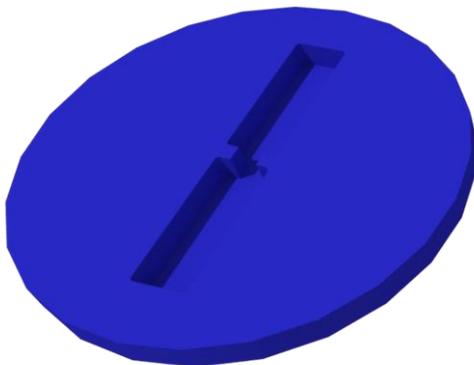
Rueggeberg FA. From vulcanite to vinyl, a history of resins in restorative dentistry. *J Prosthet Dent.* 2002;87(4):364-79.

Turssi CP, Ferracane JL e Vogel K. Filler features and their effects on wear and degree of conversion of particulate dental resin composites. *Biomaterials* 2005; 26(24): 4932-7.

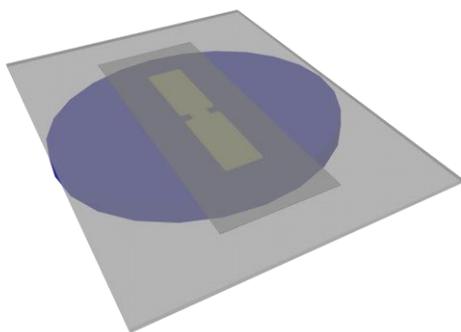
Weinmann W, Thalacker C, Guggenberger R. Siloranes in dental composites. *Dent Mater.* 2005;21(1):68-74.

## APÊNDICE

### Ilustração da metodologia para teste de resistência coesiva



- I- Molde de silicone com formato de ampulheta com 11mm em comprimento, 2mm em largura, 1mm em espessura e 1mm de largura na região mais estreita.

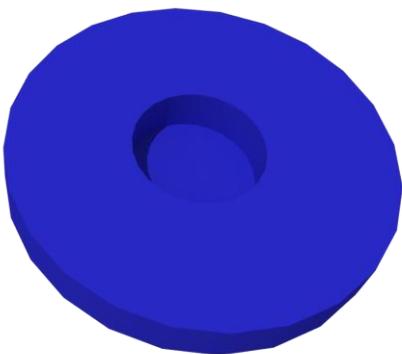


- II- Após a inserção da resina composta em um único incremento; posicionamento de tira de poliéster e placa fina de acrílico para permitir a adaptação por completo da resina composta no interior do molde.

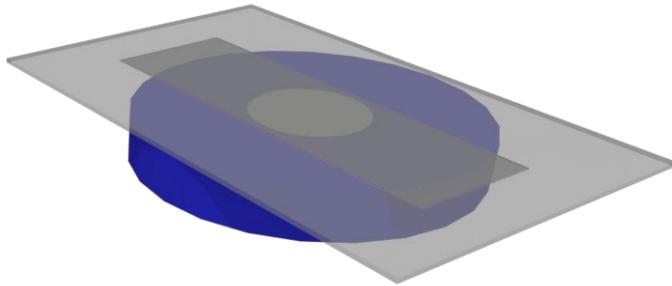


**III-** Remoção da placa fina de acrílico e fotopolimerização com ponta ativa do aparelho fotopolimerizador em contato com tira de poliéster.

**Ilustração da metodologia para teste de rugosidade de superfície e perda de massa.**



**IV-** Molde de silicone com formato circular com 5mm de diâmetro e 2mm de espessura.



**V-** Após a inserção da resina composta em um único incremento; posicionamento de tira de poliéster e placa fina de acrílico para permitir a adaptação por completo da resina composta no interior do molde.



**VI-** Remoção da placa fina de acrílico e fotopolimerização com ponta ativa do aparelho fotopolimerizador em contato com tira de poliéster.

.

Declaração do direito autoral transferido a editora quando a tese for defendida em formato alternativo



**UNICAMP**

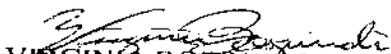
**UNIVERSIDADE ESTADUAL DE  
CAMPINAS – UNICAMP**  
**Faculdade de Odontologia de Piracicaba – FOP**



### DECLARAÇÃO

As cópias de artigos de minha autoria ou de minha co-autoria, já publicados ou submetidos para publicação em revistas científicas ou anais de congressos sujeitos a arbitragem, que constam da minha Tese de Doutorado intitulada "PROPRIEDADES MECÂNICAS E RESISTÊNCIA DA UNIÃO DE RESINAS COMPOSTAS COM MATRIZ DE SILOXANO OU COM NOPARTÍCULAS", não infringem os dispositivos da Lei nº 9.610/98, nem o direito autoral de qualquer editora.

Piracicaba, 10 de agosto de 2011.

  
VIRGINIA BOSQUIOLI

RG: 1.823.433-5

Autor(a)

MARIO ALEXANDRE COELHO SINHORETI

RG: 18.897.368

Orientador(a)