PONTIFÍCIA UNIVERSIDADE CATÓLICA DO RIO GRANDE DO SUL
FACULDADE DE ODONTOLOGIA
PROGRAMA DE PÓS-GRADUAÇÃO EM ODONTOLOGIA
ÁREA DE CONCENTRAÇÃO EM MATERIAIS DENTÁRIOS

PROPRIEDADES MECÂNICAS DE RESINAS COMPOSTAS COM NANOPARTÍCULAS

Rogério Simões Rosa

Tese apresentada como parte dos requisitos obrigatórios para a obtenção do título de Doutor em Odontologia, área de concentração em Materiais Dentários, pela Pontifícia Universidade Católica do Rio Grande do Sul.

Linha de Pesquisa: Materiais Odontológicos

Orientador: Prof. Dr. Eduardo Gonçalves Mota

Porto Alegre

2010

Dados Internacionais de Catalogação na Publicação (CIP)

R788p Rosa, Rogério Simões
Propriedades mecânicas de resinas compostas com nanopartículas / Rogério Simões Rosa. – Porto Alegre, 2010.
57f.

Tese (Doutorado) – PUCRS. Faculdade de Odontologia. Curso de Pós-Graduação em Odontologia. Área de Concentração em Materiais Dentários.
Orientador: Prof. Dr. Eduardo Gonçalves Mota.

1. Odontologia. 2. Materiais Dentários. 3. Resistência dos Materiais (Odontologia). 4. Resinas (Odontologia). 5. Nanotecnologia. I. Mota, Eduardo Gonçalves. II. Título.

CDD 617.675

Bibliotecária Responsável: Dênira Remedi – CRB 10/1779

“O valor das coisas não está no tempo que elas duram, mas na intensidade com que acontecem. Por isso, existem momentos inesquecíveis, coisas inexplicáveis e pessoas incomparáveis”.

Fernando Pessoa

Dedico esta pesquisa aos meus familiares, ao pai e colega Renato Oliveira Rosa, à mãe Maria Tereza Simões Rosa e ao irmão Rafael Simões Rosa. Assim como, à namorada Marília Zanchet, aos demais familiares, amigos, amigas e colegas que me acompanharam e me incentivaram ao longo desta etapa de minha vida.

AGRADECIMENTOS

Ao prof. Dr. José Antônio Poli de Figueiredo, coordenador do Programa de Pós-Graduação da Faculdade de Odontologia da PUCRS;

Ao meu orientador, prof. Dr. Eduardo Gonçalves Mota, coordenador do Doutorado de Materiais Dentários da Faculdade de Odontologia da PUCRS, pela dedicação em minha formação;

Aos docentes do Programa de Doutorado da Faculdade de Odontologia da PUCRS;

Aos professores da disciplina de Prótese III e IV da Faculdade de Odontologia da PUCRS pelo empenho em meu aprendizado clínico;

Aos colegas do Programa de Doutorado da sub-área de Materiais Dentários;

Aos colegas do Programa de Doutorado das sub-áreas de Dentística e Prótese Dentária.

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LISTA DE ABREVIATURAS, SIGLAS E SÍMBOLOS

LISTAGEM	DESCRIÇÃO
ADA	Associação Dentária Americana
ANOVA	Análise de Variância
BisEMA	Bisfenol A polietileno glicol diéter dimetacrilato
BisGMA	Bisfenol glicidil dimetacrilato A
cm	centímetros
°C	Grau Celsius
df	grau de liberdade
DP	Desvio padrão
EDMA	Etileno glicol dimetacrilato
F	Teste “F” (estatística)
g	grama
GPa	Gigapascal
h	hora(s)
ISO	International Organization for Standardization
kg	Quilograma
kgf	Quilograma força

KHN	Número de dureza Knoop
mg	miligrama
min	minuto
ml	mililitro
mm	milímetro
mN	mili-Newton
mol%	mol porcento
MPa	Mega Pascal
MPS	γ-Metacrilolpropilsilano
mW/cm²	mili-watt por centímetro ao quadrado
N	Newton
nm	nanômetro
ppm	parte por milhão
PTFE	Politetrafluoretileno
PVC	Polivinil Cloreto Rígido
®	marca registrada
s	segundo
p	significância
TEDMA	Trietileno dimetacrilato
TEGDA	Trietileno glicol diacrilato
TEGDMA	Trietileno glicol dimetacrilato
TEGMA	Trietileno glicol metacrilato

TGA	Análise termogravimétrica
TMPT	Trimetilol propano trimetilmetacrilato
TTEGDA	Tetraetileno glicol diacrilato
UDMA	Uretano dimetacrilato
UEDMA	Etileno uretano dimetacrilato
VHN	Número de dureza Vickers
vol%	percentual em volume
Wt%	percentual em peso
α	nível de significância
μm	micrômetro

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INTRODUÇÃO

No princípio dos anos 60, Bowen pesquisou a respeito das resinas epóxicas reforçadas com carga. Constatou deficiências em algumas propriedades do material, como a baixa velocidade de polimerização e a tendência à descoloração, que o motivaram a realizar combinações entre resinas epóxicas e acrílicas. Desse modo, ocorreu o desenvolvimento da molécula do BisGMA, a qual foi aprovada para ser utilizada como matriz para resinas compostas (BOWEN, 1963). Assim, os cimentos de silicato e as resinas acrílicas foram substituídos pelas resinas compostas em restaurações estéticas de dentes anteriores (LEINFELDER *et al.*, 1975). Na década de 70, o desenvolvimento de materiais fotopolimerizáveis abriu caminhos para novas evoluções (JACKSON e MORGAN, 2000). Estudos demonstraram que resinas compostas fotopolimerizáveis eram mais resistentes ao desgaste e apresentavam maior estabilidade de cor que as polimerizáveis quimicamente (LEINFELDER *et al.*, 1975; POWERS, FAN e RAPTIS, 1980). Foram também minimizados o tempo de presa e a inibição da polimerização pelo oxigênio ([b]ANUSAVICE, 1998). Além disso, partículas de carga de tamanhos reduzidos foram desenvolvidas, permitindo que se aumentasse seu conteúdo de carga inorgânica e, consequentemente, sua resistência mecânica (JACKSON e MORGAN, 2000). Assim, aumentaram significativamente as indicações de resinas compostas fotopolimerizáveis (TERRY, 2004).

Quanto à composição, as resinas compostas são constituídas das seguintes fases: a fase orgânica (matriz), a fase inorgânica (carga) e o agente de união (silano) (LUTZ e PHILLIPS, 1983; TERRY, 2004). Estão disponíveis em diferentes tamanhos de partículas de carga (macroparticuladas, microparticuladas, híbridas, micro-híbridas e nanoparticuladas), métodos de polimerização (quimicamente ativadas, fotopolimerizáveis e duais) e viscosidades (alto, médio ou baixo escoamento) (LUTZ e PHILLIPS, 1983; HOSODA, YAMADA e INOKOSHI, 1990; WILLEMS *et al*., 1992; LANG, JAARDA e WANG, 1992; BURGESS, WALKER e DAVIDSON, 2002).

Devido a sua resistência, estética excelente, custo acessível e sua adesividade, as resinas compostas têm sido muito utilizadas na odontologia,

sendo mais uma opção restauradora estética (LU *et al.*, 2005). Pode-se citar outras vantagens, se comparadas ao amálgama de prata, como a ausência da toxicidade do mercúrio em sua composição e o favorecimento da execução de preparos cavitários mais conservadores, devido a sua qualidade adesiva (CRAIG, POWERS e WATAHA, 2002). O aumento da demanda pela odontologia estética tem conduzido ao desenvolvimento de materiais para restaurações diretas com propriedades físicas e mecânicas melhoradas, assim como estética e durabilidade aumentadas. A última inovação neste ramo tem sido o desenvolvimento de resinas compostas nanoparticuladas, através da introdução de nanopartículas e nanoaglomerados numa matriz resinosa convencional (MITRA, WU e HOLMES, 2003).

A idéia principal da nanotecnologia não está somente na criação e na utilização de materiais a nível de átomos e moléculas, com tamanho variando de 0,1 a 100 nanômetros, mas também no aproveitamento de propriedades inerentes a estes (ZHANG *et al.,* 2005). Um nanômetro é 1/1.000 de um micrometro. Acredita-se que materiais nanoparticulados podem ser usados para produtos mais leves, mais resistentes e mais precisos. A meta é desenvolver resinas compostas que poderiam ser usadas em regiões posteriores e anteriores da boca com alto polimento inicial e com grande capacidade de retenção deste, típico das microparticuladas, assim como com propriedades mecânicas excelentes tornando-as capazes de suportar altas cargas de estresse interoclusais, típico das híbridas (MITRA, WU e HOLMES, 2003; MASOURAS, SILIKAS e WATTS, 2008; SUZUKI, 2009).

Diversas pesquisas sucederam-se, com a finalidade de avaliar outras características desse material restaurador, como o tipo de carga inorgânica incorporada a este, seu percentual em peso (LI *et al.*, 1985; NEVES *et al.*, 2002; KIM, ONG e OKUNO, 2002; MITRA, WU e HOLMES, 2003), sua silanização, o tipo de matriz orgânica e seus diluentes (ASMUSSEN e PEUTZFELDT, 1998; SHORTALL, UCTASLI e MARQUIS, 2001). Preocupando-se com sua resistência às diversas cargas que lhe são aplicadas, as propriedades mecânicas destes materiais têm sido avaliadas, como a resistência à compressão, à tração diametral (COBB *et al.*, 2000) e flexural, o módulo de elasticidade, assim como a dureza (SAY *et al.*, 2003).

Dentre as propriedades testadas neste estudo, o teste de dureza é empregado para se predizer a resistência ao desgaste de um material e sua capacidade de abrasionar estruturas dentais opostas ([a]ANUSAVICE, 1998). Para entender o comportamento de materiais restauradores, principalmente de dentes anteriores, submetidos a forças de tração é utilizado o teste de resistência à tração diametral. O módulo flexural testa a tenacidade das resinas compostas ([a]ANUSAVICE, 1998). Já o teste de resistência flexural realiza simultaneamente tensões de tração, compressão e cisalhamento no mesmo material. A aferição do percentual de carga em peso do material é importante, pois influencia em suas propriedades mecânicas (NEVES *et al.*, 2002; KIM, ONG e OKUNO, 2002). Quanto ao teste de resistência à compressão, mostra a capacidade do material de suportar o estresse funcional intra-oral.

Portanto, o objetivo desse estudo é comparar o comportamento de resinas compostas nanoparticuladas de diferentes marcas comerciais, no que se refere aos testes propostos pelo estudo. A hipótese nula é de que as resinas compostas nanoparticuladas terão desempenho semelhante nos ensaios laboratoriais realizados.

Evaluation of Mechanical Properties on three Composites with Nanoparticles

Rogério S. Rosa, Eduardo G. Mota, Hugo M. S. Oshima, Luciana Hirakata, Carlos E. A. Balbinot, Luis A. G. Pires

Pontifical Catholic University of Rio Grande do Sul, Graduate Program in Dentistry (Dental Materials). Av. Ipiranga, 6681, Building 6, Post-box: 1429, Zip code: 90619-900, Porto Alegre, Rio Grande do Sul, Brazil.

Correspondence to: Rogério S. Rosa (e-mail:rgsrosa@yahoo.com.br)

Running head: Mechanical Properties on three Composites with Nanoparticles.

Abstract: The purpose of this study was to evaluate the mechanical behavior of one nanofilled (Filtek Supreme XT - 3M ESPE) and two nanohybrid (Esthet X-Dentsply, Grandio-Voco) composites with enamel and body shades (A2) trough compressive strength test, flexural strength test, diametral tensile strength, flexural modulus, weight filler content and Knoop microhardness. Ten samples of each material were submitted to compressive strength, flexural strength and diametral tensile strength test in an universal testing machine. The flexural modulus test was calculated based on flexural strength results. Ten samples of each group were submitted to knoop microhardness test. The results were submitted to ANOVA and Tukey statistical tests. One sample of each group was used in order to register the inorganic weight filler content. An improved mechanical behavior of Grandio was observed for flexural modulus, knoop microhardness and weight filler content. For diametral tensile

strength Grandio and Filtek Supreme XT obtained higher averages. The tested composite resins ranged similar medias for compressive strength. For flexural strength Filtek Supreme XT and Esthet X showed higher results.

Keywords: composite, nanotechnology, in vitro, mechanical properties

INTRODUCTION

Resin composites are widely used in dentistry and have become one of the most commonly used esthetic restorative materials, because of their adequate strength, excellent esthetics, moderate cost and ability to bond to tooth structures. During the last few decades, the increasing demands in esthetic dentistry have led to the development of resin composite materials for direct restorations with improved physical and mechanical properties, esthetics and clinical longevity. The latest development in this field has been the introduction of nanofilled materials, by combining nanomeric particles and nanoclusters in a conventional resin matrix.[1] The essence of nanotechnology is in the development and use of materials and devices at the level of atoms and molecules with sizes of approximadly 0.02 μm, half particles size of minifilled composite resins, ranging from 0.1 to 100 nanometers and in the exploitation of these particles unique properties.[1]

The objective was to develop a composite dental filling material that could be used in anterior and/or posterior tooth restorations with high initial polish and superior polish retention, typical of microfills, as well as excellent mechanical properties, suitable for high stress bearing restorations typical of hybrid composites.[1] Nanofilled materials are believed to have high filler content,

easy handling and restoration sculpture maintainance for long time.[2] Furthermore, offer best optical properties,[1] excellent wear resistance, strength and ultimate esthetics due to their exceptional polishability, polish retention and lustrous appearance.[3] Because of reduzed nanofilled composite resins particles size and filler obtainance method, reducing polymerization shrinkage, a higher amount of filler content implies improved mechanical behavior, like diametral tensile strength, compression strength and fracture toughness, that is very important in areas with high functional stress in oral environment.[4-6]

Moreover, optimizing the adhesion of restorative biomaterials to the mineralized hard tissues of the tooth is a decisive factor for enhancing the mechanical strength, marginal adaptation and seal, in order to improve the reliability and longevity of the adhesive restoration.

The hardness test is used to relate the material wear resistance and its capacity of abrading opposite dental structures. Compressive resistance test can predict the capacity of the material to support stress functional. For understand the behavior of materials exposed to tensile stress commonly observed in anterior restorations is used diametral tensile strength test. The flexural modulus determines the composite resins relative stiffness. Flexural strength test realize simultaneously tensile, compression and shear tensions in the same material and weight filler content influence in composite resins mechanical properties.[7,8]

There are few results in literature about nanofilled composite resins mechanical properties, the objective of this in vitro study was to evaluate and to comparate the mechanical behavior of three direct composite resins different commercial brand with nanofilled or nanohybrid inorganic filler particles in

enamel and body shade (A2), trough compression strength, diametral tensile strength, three-points flexural strength, flexural modulus, Knoop microhardness and weight filler content. The null hypothesis is that these tested materials will have similar behavior in relation to mechanical properties proposed in this study.

MATERIAL AND METHODS

The composites evaluated in this study are specificated in table I.

TABLE I. Specifications of the composites evaluated in this study.[5,9]

Group and Manufacturers	Filler	Organic mould	Color	Batch number
Filtek Supreme XT (3M ESPE, St.Paul, Minessota, USA) Nanofilled	Combination of aggregated zircônia/sílica cluster with primary particle size (5-20 nm), and nonagglomerated silica filler (20 nm). 78.5 Wt%.	Bis-GMA, UDMA, TEGDMA and Bis-EMA	A2E, enamel	6BW
Grandio (VOCO, Cuxhaven, Low Saxony, Germany) Nanohybrid	Ceramic glass fine particles (1µm), spherical silicium dioxide (20-60 nm). 87.0 Wt%.	BisGMA, UDMA and TEGDMA	A2, enamel	732242
Esthet X (DENTSPLY, Milford, Delaware, USA) Nanohybrid	Barium boron fluoralumino silicate glass with particles sizes (0.6-0.8 µm) and silica nanofiller (0.04 µm). 77.0 Wt %.	Bis-GMA, Bis-EMA and TEGDMA	A2, body	070724

Compressive strength test

Compressive strength test was performed according to previous studies.[10-12] Ten samples (n=10) of each composite resin were made with 2 mm thick increments using a polytetrafluoroethylene (PTFE) mould (3 mm diameter and 6 mm height). Each increment was polymerized for 20 seconds. After the last one increment, a transparent plastic strip was positioned over the PTFE mould, and a glass slab was compressed against the mould-composite. The glass slab was removed for composite polymerization for 20s (curing unit XL–1500, 3M-ESPE, Germany, Bavaria, Seefeld) according to manufacturers recommendations. The light intensity was monitored above 400 mW/cm^2, which was monitored by a curing radiometer (model 100, Demetron/Kerr, United States of America, Connecticut, Danbury). After storage for 24 h at 37°C in an oven (model 002 CB, Fanem, Brazil, São Paulo, São Paulo), samples were placed in an universal testing machine (Emic DL 2000, Emic, Brazil, Paraná, São José dos Pinhais) at a crosshead speed of 0.50 mm/min.[12,13] Data were obtained in kgf and transformed in MPa using the following formula: $RC = F \times 9.80 / A$, where *RC* is the compressive strength (MPa), *F* is the recorded force (kgf) multiplied by the constant *9.80* (gravity), and *A* is the base area (7.06 mm²). Data were analyzed by ANOVA and Tukey`s test ($p<0.05$).

Flexural strength test

Ten samples of each composite system were made using a 25 x 2 x 2 mm metallic mould for flexural strength test.[14] The composite was packed into the metallic mould in one increment. A transparent plastic strip was positioned

over the metallic mould, and a glass slab was pressed against the mould-composite. The glass slab was removed for composite polymerization for 20 s (curing unit XL–1500, 3M-ESPE, Germany, Bavaria, Seefeld) in mould at three points. The light intensity was above 400mW/cm², which was monitored by a curing radiometer (model 100, Demetron/Kerr, United States of America, Connecticut, Danbury). The samples were stored in individual light-protected plastic tubes with distilled water at 37 ˚C for 24 hours.[13] After this step, samples were placed on a 25 mm-length supporting base and assembled in a universal testing machine (Emic DL 2000, Emic, Brazil, Paraná, São José dos Pinhais). A customized device was adapted to the upper holder to allow vertical loading of the samples according to a three-point bending test design. Axial load was applied until failure at a crosshead speed of 0.5 mm/min. Flexural strength data were obtained in kgf and transformed in MPa using the following ISO 4049 formula: $s = 3\ F\ L\ /\ 2\ b\ h^2$, where *s* is the flexural strength (MPa), *F* is the recorded force (kgf), *L* is the length between the supporting points (21 mm), *b* is the width of the prism (2 mm), and *h* is the thickness of the prism (2 mm).[14] The load-deflection curves were recorded with computer software (MTest, EMIC). Data were analyzed by ANOVA and Tukey`s test ($p<0.01$).

Flexural modulus

Based on flexural strength data, flexural modulus was calculated using the following formula: $Ef\ (GPa) = L^3\ F1\ 10^{-3}\ /\ 4b\ f\ h^3$, where *Ef* – flexural modulus; *L*– support width (mm); *F1* – load (N) at convenient point that is in straight line portion of the trace; *f* – deflection of the test sample at load *F1* (mm); *b* – breadth of the test sample (mm); and *h* – height (mm).[15] Data were analyzed by ANOVA and Tukey`s test ($p<0.01$).

Diametral tensile stength

Ten samples of each material were made using a PTFE split mould (6 mm diameter and 3 mm thickness). The composite resins were inserted in two increments and light-cured according to each manufacturer's directions. The samples were stored in distilled water at 37°C for 24 hours prior to testing.[13] After that, ten samples were mounted in a universal testing machine (Emic DL 2000, Emic, Brazil, Paraná, São José dos Pinhais) and tested with 1.00 mm/min of cross-head speed. The diametral tensile strength (MPa) was converted using the following formula*:* (2×p)/(¶×d×t)*.* Were *p* is the ultimate tensile strength (N), *d* is the diameter (6 mm) and *t* is the thickness (3 mm). Data were analyzed by ANOVA and Tukey`s test ($p<0.01$).

Knoop microhardness

Ten samples (n=10) of each composite resin were made using a PTFE split mould (6 mm diameter and 3 mm thickness). The composite resins were inserted in two increments and light-cured according to each manufacturer's directions. The samples were stored in distilled water at 37°C for 24 hours prior to testing.[13] Each sample was submitted to one indentation at knoop microhardness tester (Shimadzu HMV, Shimadzu, Japan, Kansai, Kyoto) using a load of 100 g for 15 s. Data were analyzed by ANOVA and Tukey`s test ($p<0.05$).

Weight filler content

One sample with 20 mg was made to each composite resin group. After this step, were inserted in platine crucible and submitted to temperature heating between 20-700 °C/min inside of machine for calculate the weight filler content

(TGA 2050 dispositive, TA Instruments representative, United States of America, Delaware, New Castle). The organic matrix decomposition temperature and weight filler content were registred. When stabilized sample weight, the inorganic content (Wt%) was registred.[7,8]

RESULTS

The results are summarized in Tables II and III. The compressive strength results weren`t statistically different applying ANOVA (p=0.87, Filtek Supreme XT enamel = Grandio enamel and = Esthet X body). A significant difference was observed when flexural strength (p=0.02, Filtek Supreme XT enamel > Grandio enamel and = Esthet X body), diametral tensile strength (p=0.03, Filtek Supreme XT enamel = Grandio enamel and > Esthet X body), flexural modulus (p=0.00, Grandio enamel > Filtek Supreme XT enamel > Esthet X body) and knoop microhardness (p=0.00, Grandio enamel > Filtek Supreme XT enamel > Esthet X body) of nanofilled and nanohybrids composites were compared. The compressive strength (MPa) results ranged from 184.67 (Filtek Supreme XT enamel) to 173.55 (Esthet X body). The flexural strength (MPa) results ranged from 123.29 (Filtek Supreme XT enamel) to 103.23 (Grandio enamel). The diametral tensile strength (MPa) results ranged from 50.26 (Filtek Supreme XT enamel) to 41.50 (Esthet X body). The flexural modulus (GPa) results ranged from 11.53 (Grandio enamel) to 6.46 (Esthet X body). The knoop microhardness (KHN) results ranged from 172.52 (Grandio enamel) to 54.42 (Esthet X body). The weight filler content (Wt%) were, in decrease disposition, 87.00 (Grandio enamel), 76.80 (Esthet X body) and 76.54 (Filtek Supreme XT enamel).

Table II. Compressive strength, flexural strength and flexural modulus of the tested composite resins.

	Compressive strength (MPa)		Flexural strength (MPa)		Flexural modulus (GPa)	
	Mean	SD	Mean	SD	Mean	SD
FILTEK SUPREME XT (Nanofilled)	184.67[a]	57.18	123.29[a]	21.92	8.50[b]	2.02
GRANDIO (Nanohybrid)	181.83[a]	47.77	103.23[b]	14.32	11.53[a]	1.36
ESTHET X (Nanohybrid)	173.55[a]	39.73	106.51[ab]	11.52	6.46[c]	1.39

Means followed by different letters are statistically different ($p < 0.05$).

Table III. Knoop microhardness, diametral tensile strength and weight filler content of the tested composite resins.

	Knoop microhardness (KHN)		Diametral tensile strength (MPa)		Weight filler content (Wt%)
	Mean	SD	Mean	SD	
FILTEK SUPREME XT (Nanofilled)	123.10[b]	3.51	50.26[a]	6.66	76.54
GRANDIO (Nanohybrid)	172.52[a]	76.22	42.29[ab]	9.37	87.00
ESTHET X (Nanohybrid)	54.42[c]	1.46	41.50[b]	6.94	76.80

Means followed by different letters are statistically different ($p < 0.05$).

DISCUSSION

The objective of nanotechnology is to develop a dental filling material that might be used in all areas of the mouth with high initial polish and superior

polish retention (typical of microfills), as well as excellent mechanical properties suitable for high stress bearing restorations (typical for hybrid composites).[1] The milling procedure used to make filler particles usually cannot reduce the filler particle size below 100 nm. The nanotechnology manufactures smaller filler particles with average size of 40 nm or 0.04 μm (1 μm is equal to 1000 nm in scale). The same filler size has been reached by microfilled composites since 70's. However, the real innovation that implies better mechanical behavior is the nanofiller's possibility to improve the load of the inorganic phase in 80 Wt% when compared to microfilled composites 50 Wt% for example.[16] Moreover, provides better physical, mechanical and optical properties, because these filler particles are polymerized into the resin system with molecules designed to be compatible when coupled with a polymer. There is a potential for failures in adhesion between the macroscopic (40 nm to 0.7 nm) restorative material and the nanoscopic (1nm to 10 nm in size) tooth structure, because the particle size of conventional composites are so dissimilar to the structural sizes of the hydroxyapatite crystal, dentinal tubule and enamel rod.[3] Besides, a decisive factor for enhancing the mechanical strength, marginal adaptation and seal is optimizing the adhesion of restorative biomaterials to the mineralized hard tissues of the tooth in order to improve the reliability and longevity of the adhesive restoration.

The compressive strength test is easy to perform but its interpretation is complex as tension and shear forces act concurrently inside the material. Compressive resistance cannot predict the capacity of the composite resin to support stress, and that this relationship is limited to frail materials.[12] Composite resins would suffer a "barrel" effect when submitted to a

compressive test and expand until plastic deformation occurs.[17] The results (MPa) obtained for Mitra *et al*. (2003) (Filtek Supreme Standard: 426.2, Filtek Supreme Translucent: 458.6, Esthet X: 422.1) were higher than the obtained in this study, because they used different methodology and sample size.[1] However, Mitra *et al*. (2003) results comply with the related for Filtek Supreme XT manufacturer (420 MPa).[1]

The diametral tensile strength is a mechanical property used to understand the behavior of brittle materials when exposed to tensile stress commonly observed in anterior restorations. The results (MPa) obtained in this study are similar to the average previously recorded as 44.42, 58.00 for Supreme XT, 49.24, 54.6 for Grandio enamel and 42.87 for Esthet X.[18-20] However, Mitra et al. (2003) obtained different results as 66.70 for Esthet X, 87.60 for Supreme translucent (enamel) and 80.70 for Filtek Supreme Standard (dentin), because they used ADA specification n. 27 methodology with a load of 10 mm/min.[1,21] All tested composite resins obtained higher averages than ADA specification n. 27 for direct filling resins.[21]

Restorations in functional areas are exposed to attrition and wear and microhardness may determine the abrasion resistance. The knoop microhardness (KHN) observed for Esthet X 54.42 (±1.46) comply with 54.45 (±1.47) previously registered in the dental literature validating the used methodology.[19] The media observed in this study for Supreme XT 123.10 (±3.51) was higher than 54.40 (± 2.40) previously registered in the dental literature, where the mean was determined from three measurements.[22]

The results (MPa) obtained in this study for flexural strength, that realize simultaneously tensile, compression and shear tensions, are similar to the average previously recorded as 118.00 (±12.00), 119.43 (± 18.68) for Supreme XT, 107.00 (±12.00) for Grandio enamel.[5,16] However, Da Silva, Poskus and Guimarães obtained different results as 173.70 (± 30.40) for Supreme XT (dentin), because they used a different dimensions samples light-cured without specific points and tested with 50 N load cell.[17] Yesilyurt *et al*. obtained different results as 154.40 (±29.80) for Supreme XT, because the test was done at a crosshead speed of 0.05 mm/min.[22] Júnior *et al*. and Bona *et al*. obtained different results as 145.67 (±13.96) and 119.48 (±2.10), respectly for Esthet X.[16,20] Júnior *et al*. used the universal testing machine with a crosshead speed of 1 mm/min and Bona *et al*. light-cured for 20 seconds at each third of the upper and lower surfaces of the sample.[16,20]

The flexural modulus determines the composite resins relative stiffness. The results (GPa) obtained in this study for flexural modulus are similar to the average previously recorded as 8.20 (±1.00), 8.80 (±0.70) for Supreme XT, 14.10 (±1.50) for Grandio enamel and 6.93 (± 0.69) for Esthet X.[5,16,17] However, Júnior *et al*. obtained different result as 5.76 (± 1.49) for Supreme XT (enamel and dentin), it could be because they used a crosshead speed of 1 mm/min.[16]

The composite resins weight filler content is relationed directly with their mechanical properties. The results (Wt%) obtained in this study for weight filler content are similar to the manufacturers informations as 78.50 for Supreme XT and 77.00 for Esthet X. The result (87.00 Wt%) for Grandio enamel was equal to the manufacturer information.

The null hypothesis was rejected. Weight filler content (Wt%) results could explain the different averages between groups. A high contact surface is observed between nanofillers and organic phase improving the material hardness.[19] The weight filler content is directly correlated to hardness values and a strong positive correlation ($0.88 < r < 0.96$) was registered.[8,23] Therefore, the better mechanical behavior of Grandio enamel for flexural modulus, knoop microhardness could be explained by it high weight filler percentual. For diametral tensile strength Grandio enamel and Filtek Supreme XT enamel obtained the highest results and the three tested composite resins ranged similar medias for compressive strength. For flexural strength Filtek Supreme XT enamel and Esthet X body showed the highest results.

Besides, nanoparticles have a large surface in comparison to their volume and therewith higher surface energy. Untreated, they immediately agglutinate to the usual microparticles and lose the phenomenal properties of the original nanoparticle. Therefore, it is necessary to chemically inactivate the surface of nanoparticles in order to enable their isolation.

Further in vitro and in vivo studies should evaluate the properties of nanofilled and nanohybrid composite resins.

CONCLUSIONS

In this study, improved mechanical behavior of Grandio enamel was observed for flexural modulus, knoop microhardness and weight filler content. For diametral tensile strength Grandio enamel and Filtek Supreme XT enamel obtained the best results. The tested composite resins ranged similar medias for compressive strength. For flexural strength Filtek Supreme XT enamel and

Esthet X body showed the best results. Further studies should be carried out to improve the knowledge of nanofilled and nanohybrid composites mechanical behavior.

ACKNOWLEDGMENTS

The authors would like to express appreciation to Pontifical Catholic University dental materials laboratory coordination for their technical advice and assistance.

REFERENCES

1. Mitra SB, Wu D, Holmes BN. An application of nanotechnology in advanced dental materials. J Am Dent Assoc 2003;134:1382-1390.
2. Cetin AR, Unlu N. One-year clinical evaluation of direct nanofilled and indirect composite restorations in posterior teeth. Dent Mater 2009;28:620-626.
3. Terry DA. Direct applications of a nanocomposite resin system: part 1- the evolution of contemporary composite materials. Pract Proced Aesthet Dent 2004;16:A-G.
4. Aguiar FH, Braceiro AT, Ambrosano GM, Lovadino JR. Hardness and diametral tensile strength of a hybrid composite resin polymerized with different modes and immersed in ethanol or distilled water media. Dent Mater 2005;21: 1098-1103.
5. Beun S, Glorieux T, Devaux J, Vreven J, Leloup G. Characterization of nanofilled compared to universal and microfilled composites. Dent Mater 2007;23:51-59.
6. Masouras K, Silikas N, Watts DC. Correlation of filler content and elastic properties of resin-composites. Dent Mater 2008;24:932-939.
7. Kim KH, Ong JL, Okuno O. The effect of filler loading and morphology on the mechanical properties of contemporary composites. J Prosthet Dent 2002;87: 642-649.

8. Neves AD, Discacciati JAC, Oréfice RL, Jansen WC. Correlation between degree of conversion, microhardness and inorganic content in composites. Braz Oral Res 2002;16:349-354.
9. Masotti SM, Onófrio AB, Conceição EN, Spohr AM. Uv-vis spectrophotometric direct transmittance analysis of composite resins. Dent Mater 2006;23:724-730.
10. Kildal KK, Ruyter IE. How different curing method affect mechanical properties of composites for inlays when tested in dry and wet conditions. Eur J Oral Sci 1997;105:353-361.
11. Krishnan VK, Manjusha K, Yamuna V. Effect of diluent upon the properties of a visible-light-cured dental composite. J Mater Sci Mater Med 1997;8:703-706.
12. Brosh T, Ganor Y, Belov I, Pilo R. Analysis of strength properties of light-cured resin composites. Dent Mater 1999;15:174-179.
13. Ruyter IE, Svendsen SA. Remaining metacrylate groups in composite restorative materials. Acta Odontol Scand 1978;36:75-82.
14. International Organization for Standardization. Specification of dentistry – resin-based filling materials. ISO-4049 1988.
15. Ferracane JL, Ferracane LL, Musanje L. Effect of light activation method on flexural properties of dental composites. Am J Dent 2003;16:318-322.
16. Júnior SAR, Zanchi CH, Carvalho RV, Demarco FF. Flexural strength and modulus of elasticity of different types of resin-based composites. Braz Oral Res 2007;21:16-21.
17. Da Silva EM, Poskus LT, Guimarães JG. Influence of light-polymerization modes on the degree of conversion and mechanical properties of resin composites: a comparative analysis between a hybrid and a nanofilled composite. Oper Dent 2008;33:287-293.
18. de Moraes RR, Gonçalves LS, Lancellotti AC, Consani S, Correr-Sobrinho L, Sinhoreti MA. Nanohybrid resin composites: nanofiller loaded materials or traditional microhybrid resins? Oper Dent 2009;34:551-557.
19. Mota EG, Oshima HMS, Júnior LHB, Pires LAG, Rosa RS. Evaluation of diametral tensile strength and knoop microhardness of five nanofilled composites in dentin and enamel shades. Stomatologija, Baltic Dental and Maxillofacial Journal 2006;8:67-9.

20. Bona AD, Benetti P, Borba M, Cecchetti D. Flexural and diametral tensile strength of composite resins. Braz Oral Res 2008;22:84-89.
21. New American Dental Association. Specification n. 27 for Direct Filling Resins. J Am Dent Assoc 1977;94:1191-1194.
22. Yesilyurt C, Yoldas O, Altintas SH, Kusgoz A. Effects of food-simulating liquids on the mechanical properties of a silorane based dental composite. Dent Mater 2009;28:362-367.
23. Xu HHK. Dental composite resins containing silica-fused ceramic single-crystalline whiskers with various filler levels. J Dent Res 1999;78:1304-1311.

Evaluation of Nanohardness, Elasticity Modulus and Weight Filler Content on three Composites with Nanoparticles

Rogério Simões Rosa, Eduardo Blando, Eduardo Gonçalves Mota, Hugo Mitsuo Silva Oshima, Luciana Hirakata, Roberto Hübler, Luis Antonio Gaieski Pires

Pontifical Catholic University of Rio Grande do Sul, Graduate Program in Dentistry (Dental Materials). Av. Ipiranga, 6681, Building 6, Post-box: 1429, Zip code: 90619-900, Porto Alegre, Rio Grande do Sul, Brazil.

Correspondence to: Rogério S. Rosa (e-mail:rgsrosa@yahoo.com.br)

Running head: Nanohardness and Elasticity Modulus on Composites with Nanoparticles.

Abstract: The purpose of this study was to evaluate and compare the mechanical behavior of one nanofilled (Filtek Supreme XT - 3M ESPE) and two nanohybrid (Esthet X-Dentsply, Grandio-Voco) composites with enamel and body shades (A2) trough nanohardness, elasticity modulus and weight filler content. One sample (8 mm diameter and 1 mm thickness) of each material were submitted to nanohardness and elasticity modulus in Fischrescope HV100 equipment. Five values of ten indentations were considered valids inside confidence intereval. The results were submitted to ANOVA and Tukey statistical tests (α=0.05). One sample with 20 mg was used to each composite resin group and submitted to temperature heating between 20-700 °C/min. After the organic mould decomposition the inorganic weight filler content was registered. Grandio enamel showed the best behavior for nanohardness and elasticity modulus followed by Esthet X body and Filtek Supreme XT

enamel with statistical similar behavior for elasticity modulus. For nanohardness Filtek Supreme XT enamel showed improved behavior than Esthet X body. The higher weight filler content was showed also by Grandio enamel followed by Esthet X body and Filtek Supreme XT enamel.

Keywords: composite, nanotechnology, in vitro, mechanical properties

INTRODUCTION

Resin composites are widely used in dentistry and have become one of the most commonly used esthetic restorative materials, because of their adequate strength, excellent esthetics, moderate cost and ability to bond to tooth structures.[1] During the last few decades, the increasing demands in esthetic dentistry have led to the development of resin composite materials for direct restorations with improved physical and mechanical properties, esthetics and durability.[2,3] The latest development in the field has been the introduction of nanofilled materials, by combining nanomeric particles and nanoclusters in a conventional resin matrix.[4] The essence of nanotechnology is in the development and use of materials and devices at the level of atoms and molecules with sizes of approximadly 0.02 μm, minifilled composite resins particles size half, ranging from 0.1 to 100 nanometers and in the exploitation of these particles unique properties.[4]

The objective was to develop a composite dental filling material that could be used in all areas of the mouth with high initial polish and superior polish retention (typical of microfills), as well as excellent mechanical properties, suitable for high stress bearing restorations (typical of hybrid composites).[4] Nanofilled materials are believed to have high filler content, easy handling and

restoration sculpture maintainance for long time.[5] Furthermore, offer best optical properties,[4] excellent wear resistance, strength and ultimate esthetics due to their exceptional polishability, polish retention and lustrous appearance.[6] Because of reduzed nanofilled composite resins particles size and filler obtainance method, reducing polymerization shrinkage, a higher amount of filler content implies better mechanical behavior, that is very important in areas high stress functional in oral environment.[7-9]

Moreover, optimizing the adhesion of restorative biomaterials to the mineralized hard tissues of the tooth is a decisive factor for enhancing the mechanical strength, marginal adaptation and seal, in order to improve the reliability and longevity of the adhesive restoration.

The hardness test is used to relate the material wear resistance and to expound its capacity of abrading opposite dental structures. The elasticity modulus will describe the composite resins relative stiffness and the weight filler content influence in their mechanical properties.[10,11]

There are few results in literature about nanofilled composite resins mechanical properties, the objective of this in vitro study was to evaluate and to comparate the mechanical behavior of three direct composite resins different commercial brand with similar sizes of inorganic filler particles (nanofilled or nanohybrid) in enamel and body shade (A2) trough nanohardness, elasticity modulus and weight filler content. The null hypothesis is that these tested materials will have similar behavior in relation to mechanical properties proposed in this study.

MATERIAL AND METHODS

The composites evaluated in this study are specificated in table I.

TABLE I. Specifications of the composites evaluated in this study.[8,12]

Group and Manufacturers	Filler	Organic mould	Color	Batch number
Filtek Supreme XT (3M ESPE, St Paul, Minessota, USA) Nanofilled	Combination of aggregated zircônia/sílica cluster with primary particle size (5-20 nm), and nonagglomerated silica filler (20 nm). 78.5 Wt%.	Bis-GMA, UDMA, TEGDMA and Bis-EMA	A2E, enamel	6BW
Grandio (Voco,Cuxhaven, Low Saxony, Germany) Nanohybrid	Ceramic glass fine particles (1µm), spherical silicium dioxide (20-60 nm). 87.0 Wt%.	BisGMA, UDMA and TEGDMA	A2, enamel	732242
Esthet X (Dentsply, Milford, Delaware, USA) Nanohybrid	Barium boron fluoralumino silicate glass with particles sizes (0.6-0.8 µm) and silica nanofiller (0.04 µm). 77.0 Wt%.	Bis-GMA, Bis-EMA and TEGDMA	A2, body	070724

Nanohardness and Elasticity modulus

One sample of each composite resin were made using a mould with diameter 8 mm and height 1mm central hole. The composite was packed into the central hole in three 2 mm increments. A transparent plastic strip was positioned over the mould, and a glass slab was pressed against the mould-composite. The glass slab was removed for initial composite polymerization for

20 s (curing unit XL–1500, 3M-ESPE, Germany, Bavaria, Seefeld) with light intensity between 400-600 mW/cm², which was monitored by a radiometer (model 100, Demetron/Kerr, United States of America, Connecticut, Danbury). After this step, the samples were removed from the mould and tested in nanohardness equipment (Fischerscope HV 100, Fischer, Germany, Baden-Württemberg, Sindelfingen) for elastic, plastic and mechanic properties analysis. Ten indentations were made in each sample with Berckovich indentator. However, were considered a minimal of 5 valid values into the confidence interval. A dynamic load-unload cycle, with load graduated increase and decrease, was applied in 40 seconds to each sample. The maximum load applied in samples was 500 mN. After the nanohardness test, the load and the corresponding deflection were recorded and used to calculate the elasticity modulus (GPa).[13] Data were analyzed by ANOVA and Tukey`s test ($p<0.05$).

Weight filler content

One sample with 20 mg was used to each composite resin group. The samples were inserted in platine crucible and submitted to temperature heating between 20-700 °C/min in TGA 2050 dispositive (TA Instruments, EUA, New Castle, DW, USA). The organic mould decomposition temperature and inorganic weight filler content were registred. When stabilized sample weight, the inorganic content was registred.[10,11]

RESULTS

The results are summarized in Table II. A significant difference was observed when nanofilled and nanohybrid composites nanohardness ($p=0.00$, Grandio enamel > Filtek Supreme XT enamel > Esthet X body) and elasticity

modulus results (p=0.00, Grandio enamel > and Filtek Supreme XT enamel = Esthet X body) were compared. The nanohardness (MPa) results ranged from 727.01 (Grandio enamel) to 392.94 (Esthet X body). The elasticity modulus (GPa) results ranged from 19.78 (Grandio enamel) to 12.30 (Esthet X body).

The weight filler content (Wt%) results were, in decrease disposition, 87.00 (Grandio enamel), 76.80 (Esthet X body) and 76.54 (Filtek Supreme XT enamel).

TABLE II. Nanohardness, elasticity modulus and weight filler content of the tested composite resins.

	Nanohardness (MPa)		Elasticity modulus (GPa)		Weight filler content (wt%)
	Mean	SD	Mean	SD	
FILTEK SUPREME XT (Nanofilled)	474.79[b]	21.77	12.77[b]	0.89	76.54
GRANDIO (Nanohybrid)	727.01[a]	21.55	19.78[a]	1.51	87.00
ESTHET X (Nanohybrid)	392.94[c]	25.88	12.30[b]	0.40	76.80

Means followed by different letters are statistically different ($p < 0.05$)

DISCUSSION

The objective of nanotechnology is to develop a dental filling material that might be used in all areas of the mouth with high initial polish and superior polish retention (typical of microfills), as well as excellent mechanical properties suitable for high stress bearing restorations (typical for hybrid composites).[4] The milling procedure used to make filler particles usually cannot reduce the

filler particle size below 100 nm. The nanotechnology manufactures smaller filler particles with average size of 40 nm or 0.04 μm, because 1 μm is equal to 1000 nm in scale. The same filler size has been reached by microfilled composites since 70's. However, the real innovation that implies better mechanical behavior is the nanofiller's possibility to improve the load of the inorganic phase in 80 Wt% when compared to microfilled composites 50 Wt% for example.[14] Moreover, provides improved physical, mechanical and optical properties, because these filler particles are polymerized into the resin system with molecules designed to be compatible when coupled with a polymer. There is a potential for failures in adhesion between the macroscopic (40 nm to 0.7 nm) restorative material and the nanoscopic (1nm to 10 nm in size) tooth structure, because the particle size of conventional composites are so dissimilar to the structural sizes of the hydroxyapatite crystal, dentinal tubule, and enamel rod.[15] Besides, a decisive factor for enhancing the mechanical strength, marginal adaptation and seal is optimizing the adhesion of restorative biomaterials to the mineralized hard tissues of the tooth in order to improve the reliability and longevity of the adhesive restoration.

Restorations in functional areas are exposed to attrition and wear, then the hardness may determine the abrasion resistance. How the filler is very small, nanohardness was applied in order to record the behavior in a minor area. This test was realized with Fischerscope equipment that permits realization of indentation dynamic tests and application of desired load direct tests. The dynamic tests can be realized in 0.4 to 1000 mN load scale with possibility of 1 to 999 load steps number. The Fischerscope equipment controls load step application period in 0.1 to 999 seconds time intereval, allowing load

test speed variation, what isn`t possible in microhardness test where a unique load value is applied for determined time without the possibility of load time intereval regulation.

The elasticity modulus determines the composite resins relative stiffness. Nanohardness test give little information about the bulk of the material, because of the limited depths of penetration and the small loads applied. Thus, elasticity modulus values must be examined in conjunction with the microstructure of the material's surface.[16] The results (GPa) obtained in this study for elasticity modulus are similar to the average previously recorded as 12.40, 12.70 for Supreme XT (12.77 ± 0.89) and 20.20, 20.40 for Grandio (19.78 ± 1.51).[17,18] Besides, the media previously recorded ranged from 9.31 to 12.54 GPa for spherical fillers model dental resin-composites and from 14.09 to 17.03 GPa for irregular fillers.[16]

However, some results (GPa) are different to the average previously recorded as 8.2 (±1.00), 5.76 (± 1.49), for Supreme XT (12.77 ± 0.89),[8,14] 14.10 (±1.50) for Grandio enamel (19.78 ± 1.51) and 6.93 (± 0.69) for Esthet X (12.30 ± 0.40).[8,14] The obtained results were differents, because was used the Fischerscope HV 100 nanohardness equipment with dynamic load-unload cycle. The load graduated increase and decrease was applied in 120 seconds total time (40 seconds to each sample) and the maximum load applied in samples was 500 mN.

The composite resins weight filler content is relationed directly with their mechanical properties. The results (Wt%) obtained in this study for weight filler content are similar to the manufacturers information as 78.50 for Supreme XT (76.54), and 77.00 for Esthet X (76.80). The result (Wt%) was equal to the

manufacturer information for Grandio (87.00). The results (Wt%) are similar to the average previously recorded as 71.90 for Supreme XT and 84.10 for Grandio.[8]

The null hypothesis was rejected. Weight filler content (Wt%) results could explain the different averages between groups. A high contact surface is observed between nanofillers and organic phase improving the material hardness.[19] The weight filler content is directly correlated to hardness values and a strong positive correlation ($0.88 < r < 0.96$) was registered.[10,20] Therefore, the higher averages hardness test and elasticity modulus values observed with Grandio could be explained by it high filler content.

Besides, nanoparticles have a higher surface energy. It is necessary to chemically inactivate the surface of nanoparticles in order to enable their isolation. The filler size and shape of composite resins seemed to be a fine tuning factor for the determination of elasticity modulus. Larger filler sizes tend to render the material stiffer and irregular filler shapes result in higher modulus values than resin composites with spherical fillers.[16]

Further in vitro and in vivo studies should evaluate the properties of nanofilled and nanohybrid composite resins.

CONCLUSIONS

In this study, Grandio enamel showed improved behavior for nanohardness and elasticity modulus followed by Esthet X body and Filtek Supreme XT enamel with statistical similar behavior for elasticity modulus. For nanohardness Filtek Supreme XT enamel showed improved behavior in relation to Esthet X body. The higher weight filler content was showed also by Grandio

enamel followed by Esthet X body and Filtek Supreme XT enamel. Further studies should be carried out to improve the knowledge of the mechanical behavior of nanofilled and nanohybrid composites.

ACKNOWLEDGMENTS

The authors would like to express appreciation to Pontifical Catholic University dental materials laboratory coordination for their technical advice and assistance.

REFERENCES

1. de Moraes RR, Gonçalves LS, Lancellotti AC, Consani S, Correr-Sobrinho L, Sinhoreti MA. Nanohybrid resin composites: nanofiller loaded materials or traditional microhybrid resins? Oper Dent 2009;34:551-557.

2. Curtis AR, Palin WM, Fleming GJP, Shortall ACC, Marquis PM. The mechanical properties of nanofilled resin-based composites: The impact of dry and wet cyclic pre-loading on bi-axial flexure strength. Dent Mater 2009;25:188-197

3. Clifford SS, Roman-Alicea K, Tantbirojn D, Versluis A. Shrinkage and hardness of dental composites acquired with different curing light sources. Quintessence Int 2009; 40:203-214

4. Mitra SB, Wu D, Holmes BN. An application of nanotechnology in advanced dental materials. J Am Dent Assoc 2003;134:1382-1390.

5. Cetin AR, Unlu N. One-year clinical evaluation of direct nanofilled and indirect composite restorations in posterior teeth. Dent Mater 2009;28:620-626.

6. Suzuki T, Kyoizumi H, Finger WJ, Kanehira M, Endo T, Utterodt A, Hisamitsu H, Komatsu M. Resistance of nanofill and nanohybrid resin composites to toothbrush abrasion with calcium carbonate slurry. Dent Mater 2009;28:708-716.

7. Aguiar FH, Braceiro AT, Ambrosano GM, Lovadino JR. Hardness and diametral tensile strength of a hybrid composite resin polymerized with different modes and immersed in ethanol or distilled water media. Dent Mater 2005;21:1098-1103.

8. Beun S, Glorieux T, Devaux J, Vreven J, Leloup G. Characterization of nanofilled compared to universal and microfilled composites. Dent Mater 2007;23:51-59.
9. Masouras K, Silikas N, Watts DC. Correlation of filler content and elastic properties of resin-composites. Dent Mater 2008;24:932-939.
10. Neves AD, Discacciati JAC, Oréfice RL, Jansen WC. Correlation between degree of conversion, microhardness and inorganic content in composites. Braz Oral Res 2002;16:349-354.
11. Kim KH, Ong JL, Okuno O. The effect of filler loading and morphology on the mechanical properties of contemporary composites. J Prosthet Dent 2002;87:642-649.
12. Masotti SM, Onófrio AB, Conceição EN, Spohr AM. Uv-vis spectrophotometric direct transmittance analysis of composite resins. Dent Mater 2006;23:724-730.
13. Vasconcellos LA, Blando E, Souto AA, Oliveira MG, Woitchunas GFP, Hubler R. Study of mechanical degradation of UHMWPE acetabular components due to clinical X-ray procedures. J Mater Sci: Mater Med 2007;18:1659-1663.
14. Júnior SAR, Zanchi CH, Carvalho RV, Demarco FF. Flexural strength and modulus of elasticity of different types of resin-based composites. Braz Oral Res 2007;21:16-21.
15. Terry DA. Direct applications of a nanocomposite resin system: part 1- the evolution of contemporary composite materials. Pract Proced Aesthet Dent 2004;16:A-G.
16. Masouras K, Akhtar R, Watts DC, Silikas N. Effect of filler size and shape on local nanoindentation modulus of resin-composites. J Mater Sci: Mater Med 2008;19:3561-3566
17. Papadogiannis DY, Lakes RS, Papadogiannis Y, Palaghias G, Helvatjoglu-Antoniades M. The effect of temperature on the viscoelastic properties of nano-hybrid composites. Dent Mater 2008;24:257-266
18. Yamanel K, Çaglar A, Gülsahi K, Özden UA. Effects of different ceramic and composite materials on stress distribution in inlay and onlay cavities: 3-D finite element analysis. Dent Mater 2009;28:661-670

19. Mota EG, Oshima HMS, Júnior LHB, Pires LAG, Rosa RS. Evaluation of diametral tensile strength and knoop microhardness of five nanofilled composites in dentin and enamel shades. Stomatologija, Baltic Dental and Maxillofacial Journal 2006;8:67-9
20. Xu HHK. Dental composite resins containing silica-fused ceramic single-crystalline whiskers with various filler levels. J Dent Res 1999;78:1304-1311

DISCUSSÃO

A demanda maior pela odontologia estética tem conduzido ao desenvolvimento de materiais para restaurações diretas com melhores propriedades físicas e mecânicas, assim como maior qualidade estética e durabilidade. Embora existam muitas pesquisas nesta área, as resinas compostas ainda apresentam algumas desvantagens, como a contração de polimerização e os processos complexos envolvidos nos seus mecanismos de desgaste. Quando utilizadas em dentes posteriores, devem apresentar uma dureza inicialmente alta e satisfatória com o passar do tempo, sendo essa razoável indicadora da quantidade de desgaste. Essa resistência está relacionada com sua capacidade de abrasionar estruturas dentais opostas ([a]ANUSAVICE, 1998).

Torna-se importante conceituar propriedades mecânicas, para melhor compreensão dos testes descritos nesta tese. Referem-se à habilidade do material de resistir a forças aplicadas (cargas) sem que haja fratura ou deformação excessiva ([a]ANUSAVICE, 1998). São definidas pelas leis da mecânica, isto é, a ciência física que lida com a energia, forças e seus efeitos nos corpos principalmente estáticos ([a]ANUSAVICE, 1998).

A meta da nanotecnologia foi desenvolver resinas compostas que poderiam ser usadas em regiões posteriores e anteriores da boca com alto polimento inicial e com grande capacidade de retenção deste, típico das microparticuladas, assim como com propriedades mecânicas excelentes tornando-as capazes de suportar altas cargas de estresse interoclusais, típico das micro-híbridas (MITRA, WU e HOLMES, 2003). As resinas compostas nanoparticuladas são capazes de oferecer excelente resistência ao desgaste, resistência e estética, devido a sua excelente capacidade de polimento, retenção deste e aparência lustrosa (MITRA, WU e HOLMES, 2003).

Dentre os critérios de inclusão desta pesquisa esteve o tamanho das partículas de carga das resinas compostas em teste (nanoparticuladas ou nano-híbridas), visto que essas, em geral, diferenciam-se principalmente pela sua matriz inorgânica (BASEREN, 2004), que se apresenta disposta em

nanopartículas e nanoaglomerados numa matriz orgânica convencional. As resinas compostas nanoparticuladas apresentam somente partículas esféricas de tamanho nanométrico, ao passo que as nanohíbridas contêm partículas irregulares microhíbridas além de nanopartículas. Além disso, utilizou-se as resinas compostas com cores de esmalte ou de corpo, já que algumas pesquisas tiveram como resultado desempenhos semelhantes para estes dois tipos de materiais restauradores (MOTA, 2005). Definiu-se pela cor A2 para todas as amostras, uma vez que a variação da cor pode influir nos valores das propriedades mecânicas testadas (SAAD *et al.*, 2004).

No que se refere às propriedades testadas neste estudo, o teste de dureza é empregado para se predizer a resistência ao desgaste de um material e sua capacidade de abrasionar estruturas dentais opostas ([a]ANUSAVICE, 1998). Para entender o comportamento de materiais restauradores, principalmente de dentes anteriores, submetidos a forças de tração é utilizado o teste de resistência à tração diametral. O módulo flexural testa a tenacidade das resinas compostas ([a]ANUSAVICE, 1998). Já o teste de resistência flexural realiza simultaneamente tensões de tração, compressão e cisalhamento no mesmo material. A aferição do percentual de carga em peso do material é importante, pois influencia em suas propriedades mecânicas (NEVES *et al.*, 2002; KIM, ONG e OKUNO, 2002). Quanto ao teste de resistência à compressão, mostra a capacidade do material de suportar o estresse funcional intra-oral.

Antes da realização dos testes laboratoriais, fez-se um delineamento estatístico para que esta análise estivesse de acordo com a metodologia a ser aplicada.

Observou-se que a resina composta Grandio apresentou elevado valor de desvio-padão (76,22) no teste de microdureza Knoop quando comparado aos dos demais grupos. Acredita-se que a possível presença de pequenas bolhas nas amostras no momento da confecção das mesmas, a área escolhida na amostra para se fazer a indentação e outros fatores relacionados com a metodologia possam explicar este fato.

Constata-se que as médias de microdureza Knoop das resinas compostas testadas neste trabalho foram relativamente baixas, se comparadas com a do esmalte dental (343,00 KHN). Quando comparadas com a média da maioria das marcas comerciais de amálgama (110,00 KHN), observa-se que Grandio e Filtek Supreme XT apresentaram médias maiores (172,52 e 123,10 KHN, respectivamente) e Esthet X apresentou média menor (54,42 KHN). Quanto à resistência flexural, a média da Filtek Supreme XT (123,29 MPa) foi ligeiramente maior do que a do amálgama de fase dispersa Dispersalloy da Dentsply (122,00 MPa). No que se refere à resistência à compressão, as médias das resinas compostas testadas neste trabalho foram relativamente baixas se comparadas com a do esmalte dental (384,00 MPa), da dentina (297,00 MPa) e do amálgama de fase dispersa Dispersalloy da Dentsply (423,00 MPa) (REIS A. e LOGUÉRCIO A.D., 2009).

A hipótese nula de que as resinas compostas nanohíbridas e a nanoparticulada teriam comportamento mecânico similar nos testes propostos por este estudo foi rejeitada. O alto conteúdo de carga em peso (Wt%) da resina composta Grandio e a grande superfície de contato existente entre suas nanopartículas e a matriz orgânica circundante podem explicar suas médias mais altas, após a realização de uma análise estatística, para as propriedades mecânicas, tais como dureza e módulo de elasticidade (NEVES *et al*., 2002; MOTA, 2005).

Sugere-se que outros estudos laboratoriais e clínicos sejam realizados e que outras propriedades sejam avaliadas também, tais como rugosidade superficial antes e depois da abrasão por escovação, solubilidade, absorção de água e grau de conversão.

REFERÊNCIAS [♦]

3M ESPE. *Filtek Supreme XT Universal Restorative System. Technical Product Profile*. St. Paul, 2005.

[a]ANUSAVICE, K.J. Propriedades mecânicas dos materiais dentários. In: Anusavice, K.J. **Phillips materiais dentários**. Rio de Janeiro: Guanabara Koogan, 1998. Cap. 4, p. 28-43.

[b]ANUSAVICE, K.J. Resinas para restauração. In: Anusavice, K.J. **Phillips materiais dentários**. Rio de Janeiro: Guanabara Koogan, 1998. Cap. 12, p. 161-177.

ASMUSSEN, E.; PEUTZFELDT, A. Influence of UEDMA, BISGMA and TEGDMA on selected mechanical properties of experimental resin composites. **Dent Mater**, Copenhagen, v. 14, n. 1, p. 51-56, Jan. 1998.

BASEREN, M. Surface roughness of nanofill and nanohybrid composite resin and ormocer-based tooth-colored restorative materials after several finishing and polishing procedures. **J Biomater Appl**, London, v. 19, n. 2, p.121-134, Oct. 2004.

BOWEN, R.L. Properties of a silica-reinforced polymer for dental restorations. **J Am Dent Assoc**, Washington, v. 66, p. 57-64, Jan. 1963.

BURGESS, J.O.; WALKER, R.; DAVIDSON, J.M. Posterior resin-based composite: review of the literature. **Pediatr Dent**, Chicago , v. 24, n. 5, p. 465-479, Sep./Oct. 2002.

COBB, D.S. *et al.* The phisical properties of packable and conventional posterior resin-based composites: a comparison. **J Am Dent Assoc**, Washington, v. 131, n. 11, p. 1610-1615, Nov. 2000.

CRAIG, R.G.; POWERS, J.M.; WATAHA, J.C. Materiais para restaurações estéticas diretas. In: Craig, R.G.; Powers, J.M.; Wataha, J.C. **Materiais Dentários: Propriedades e Manipulação.** 7[a] ed. São Paulo: Ed. Santos, 2002. Cap. 4, p. 57-78.

HOSODA, H.; YAMADA, T.; INOKOSHI, S. SEM and elemental analysis of composite resins. **J Prosthet Dent**, St. Louis, v. 64, n. 6, p. 669-676, Dec. 1990.

JACKSON, R.D.; MORGAN, M. The new posterior resins and a simplified placement technique. **J Am Dent Assoc**, Washington, v. 131, n. 3, p. 375-383, Mar. 2000.

[♦] De acordo com a NBR 6023 de agosto de 2002.

KIM, K.H.; ONG, J.L.; OKUNO, O. The effect of filler loading and morphology on the mechanical properties of contemporary composites. **J Prosthet Dent**, St. Louis, v. 87, n. 6, p. 642-649, June 2002.

LANG, B.R.; JAARDA, M.; WANG, R.F. Filler particle size and composite resin classification systems. **J Oral Rehabil**, Oxford, v. 19, n. 4, p. 569-584, July 1992.

LEINFELDER, K.F. *et al.* Clinical evaluation of composite resins as anterior and posterior restorative materials. **J Prosthet Dent**, St. Louis, v. 33, n. 4, p. 407-416, Apr. 1975.

LI, Y. *et al.* Effect of filler content and size on properties of composites. **J Dent Res**, Chicago, v. 64, n. 12, p. 1396-1401, Dec. 1985.

LU, H. *et al.* Effect of surface roughness on stain resistance of dental resin composites. **J Esthet Restor Dent**, Hamilton, v. 17, n. 2, p. 102-109, 2005.

LUTZ, F.; PHILLIPS, R.W. A classification and evaluation of composite resin systems. **J Prosthet Dent**, St. Louis, v. 50, n. 4, p. 480-488, Oct. 1983.

MASOURAS, K.; SILIKAS, N.; WATTS D.C. Correlation of filler content and elastic properties of resin-composites. **Dent Mater**, Kidlington, v. 24, n. 7, p. 932–939, Nov. 2008.

MITRA, S.B.; WU, D.; HOLMES, B. An application of nanotechnology in advanced dental materials. **J Am Dent Assoc**, Washington, v. 134, n. 10, p. 1382-1390, Oct. 2003.

MOTA, E.G. **Resinas compostas: comparação de propriedades mecânicas**. 2005. 130f. Tese (Doutorado em Odontologia) – Faculdade de Odontologia, Pontifícia Universidade Católica do Rio Grande do Sul, Porto Alegre, 2005.

NEVES, A.D. *et al.* Correlação entre grau de conversão, microdureza e conteúdo inorgânico em compósitos. **Pesqui Odontol Bras**, São Paulo, v. 16, n. 4, p. 349-354, out./dez. 2002.

POWERS, J.M.; FAN, P.L.; RAPTIS, C.N. Color stability of new composite restorative materials under accelerated aging. **J Dent Res**, Chicago, v. 59, n. 12, p. 2071-2074, Dec. 1980.

REIS, A.; LOGUERCIO, A.D. Resinas compostas. In: Reis, A.; Loguercio, A.D. **Materiais dentários diretos-dos fundamentos à aplicação clínica**. São Paulo: Editora Santos, 2009. Cap. 5, p. 137-180.

SAAD, J.R.C. *et al.* Avaliação da microdureza de compostos à base de resina em função da cor e da profundidade. **J Bras Clin Odontol Int**, Curitiba, v. 8, n. 47, p. 415-418, out./dez. 2004.

SAY, E.C. *et al.* Wear and microhardness of different resin composite materials. **Oper Dent**, Seattle, v. 28, n. 5, p. 628-634, Sep. 2003.

SHORTALL, A.C.; UCTASLI, S.; MARQUIS, P.M. Fracture resistance of anterior, posterior and universal light activated composite restoratives. **Oper Dent**, Seattle, v. 26, n. 1, p. 87-96, Jan./Feb. 2001.

SUZUKI, T. *et al.* Resistance of nanofill and nanohybrid resin composites to toothbrush abrasion with calcium carbonate slurry. **Dent Mat,** Kidlington, v. 28, n. 6, p. 708–716, Nov. 2009.

TERRY, D.A. Direct applications of a nanocomposite resin system: part 1- the evolution of contemporary composite materials. **Pract Proced Aesthet Dent**, Mahwah, v. 16, n. 6, p. A-G, July 2004.

WILLEMS, G. *et al.* A classification of dental composites according to their morphological and mechanical characteristics. **Dent Mater**, Copenhagen, v. 8, n. 6, p. 310-319, Sep. 1992.

ZHANG, Y. *et al*. Recent development of polimer nanofibers for biomedical and biotechnological applications. **J Mater Sci Mater Med**, London, v. 16, n. 10, p. 933-946, Oct. 2005.

ANEXOS

ANÁLISE ESTATÍSTICA

Tests of Normality

		Kolmogorov-Smirnov(a)			Shapiro-Wilk		
	RESINA	Statistic	df	Sig.	Statistic	df	Sig.
Comp MPa	1 = Supreme XT A2E	,166	10	,200(*)	,960	10	,780
	2 = Esthet X	,265	10	,046	,867	10	,093
	3 =Grandio	,191	10	,200(*)	,904	10	,244
TD MPa	1 = Supreme XT A2E	,157	10	,200(*)	,926	10	,414
	2 = Esthet X	,157	10	,200(*)	,978	10	,954
	3 =Grandio	,221	10	,183	,858	10	,073
F MPa	1 = Supreme XT A2E	,182	10	,200(*)	,882	10	,136
	2 = Esthet X	,131	10	,200(*)	,940	10	,558
	3 =Grandio	,140	10	,200(*)	,936	10	,514
MF GPa	1 = Supreme XT A2E	,239	10	,110	,923	10	,382
	2 = Esthet X	,132	10	,200(*)	,990	10	,997
	3 =Grandio	,207	10	,200(*)	,866	10	,090

* This is a lower bound of the true significance.

a Lilliefors Significance Correction

Test of Homogeneity of Variances

	Levene Statistic	df1	df2	Sig.
Comp MPa	,237	2	27	,790
TD MPa	,678	2	27	,516
F MPa	4,661	2	27	,018
MF GPa	,422	2	27	,660

ANOVA

		Sum of Squares	df	Mean Square	F	Sig.
Comp MPa	Between Groups	667,595	2	333,797	,140	,870
	Within Groups	64183,307	27	2377,160		
	Total	64850,902	29			
TD MPa	Between Groups	469,609	2	234,804	3,900	,033
	Within Groups	1625,373	27	60,199		
	Total	2094,982	29			
F MPa	Between Groups	2315,768	2	1157,884	4,243	,025
	Within Groups	7367,819	27	272,882		
	Total	9683,587	29			
MF GPa	Between Groups	130,158	2	65,079	24,642	,000
	Within Groups	71,305	27	2,641		
	Total	201,463	29			

Comp MPa

Tukey HSD[a]

RESINA	N	Subset for alpha = .05
		1
2 = Esthet X	10	173,550
3 =Grandio	10	181,830
1 = Supreme XT A2E	10	184,670
Sig.		,867

Means for groups in homogeneous subsets are displayed.

a. Uses Harmonic Mean Sample Size = 10,000.

TD MPa

Tukey HSD[a]

RESINA	N	Subset for alpha = .05	
		1	2
2 = Esthet X	10	41,500	
3 =Grandio	10	42,290	42,290
1 = Supreme XT A2E	10		50,260
Sig.		,972	,073

Means for groups in homogeneous subsets are displayed.

a. Uses Harmonic Mean Sample Size = 10,000.

F MPa

Tukey HSD[a]

RESINA	N	Subset for alpha = .05	
		1	2
3 =Grandio	10	103,230	
2 = Esthet X	10	106,510	106,510
1 = Supreme XT A2E	10		123,290
Sig.		,897	,077

Means for groups in homogeneous subsets are displayed.

a. Uses Harmonic Mean Sample Size = 10,000.

MF GPa

Tukey HSD[a]

RESINA	N	Subset for alpha = .05		
		1	2	3
2 = Esthet X	10	6,460		
1 = Supreme XT A2E	10		8,500	
3 =Grandio	10			11,530
Sig.		1,000	1,000	1,000

Means for groups in homogeneous subsets are displayed.

a. Uses Harmonic Mean Sample Size = 10,000.

Tests of Normality

		Kolmogorov-Smirnov(a)			Shapiro-Wilk		
	RESINA	Statistic	df	Sig.	Statistic	df	Sig.
KNOOP	1 = Supreme XT A2E	,167	10	,200(*)	,936	10	,505
	2 = Esthet X	,107	10	,200(*)	,988	10	,994
	3 =Grandio	,280	10	,025	,841	10	,046

* This is a lower bound of the true significance.

a Lilliefors Significance Correction

Test of Homogeneity of Variances

KNOOP

Levene Statistic	df1	df2	Sig.
40,185	2	27	,000

ANOVA

KNOOP

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	70356,296	2	35178,148	18,119	,000
Within Groups	52420,612	27	1941,504		
Total	122776,9	29			

KNOOP

	RESINA	N	Subset for alpha = .05		
			1	2	3
Tukey HSD[a]	2 = Esthet X	10	54,4200		
	1 = Supreme XT A2E	10		123,1000	
	3 =Grandio	10			172,5200
	Sig.		1,000	1,000	1,000

Means for groups in homogeneous subsets are displayed.

a. Uses Harmonic Mean Sample Size = 10,000.

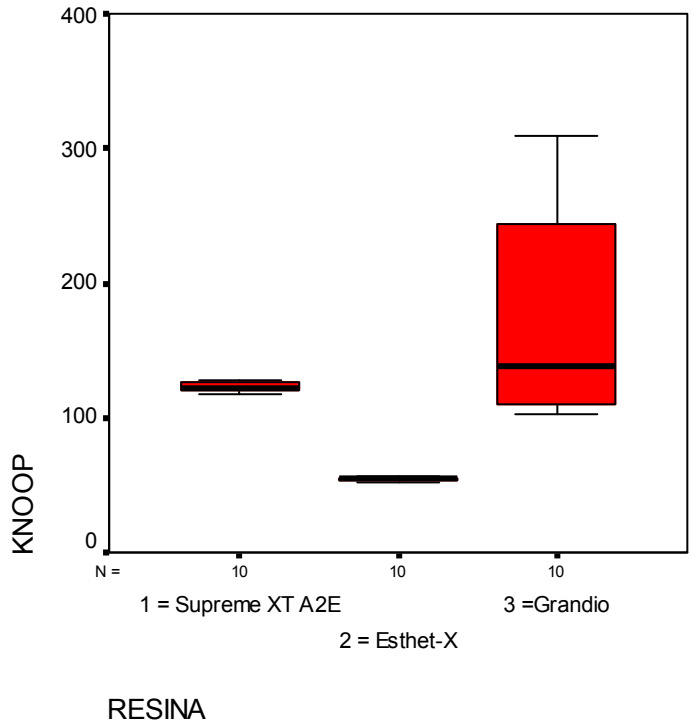
Multiple Comparisons

Dependent Variable: KNOOP

Games-Howell

(I) RESINA	(J) RESINA	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
					Lower Bound	Upper Bound
1 = Supreme XT A2E	2 = Esthet X	68,6800*	1,20617	,000	65,4674	71,8926
	3 =Grandio	-49,4200	24,12941	,156	-116,7371	17,8971
2 = Esthet X	1 = Supreme XT A2E	-68,6800*	1,20617	,000	-71,8926	-65,4674
	3 =Grandio	-118,1000*	24,10848	,002	-185,4015	-50,7985
3 =Grandio	1 = Supreme XT A2E	49,4200	24,12941	,156	-17,8971	116,7371
	2 = Esthet X	118,1000*	24,10848	,002	50,7985	185,4015

*. The mean difference is significant at the .05 level.



Tests of Normality

		Kolmogorov-Smirnov[a]			Shapiro-Wilk		
	RESINA	Statistic	df	Sig.	Statistic	df	Sig.
NANOD	Supreme	,230	5	,200*	,888	5	,348
	Grandio	,216	5	,200*	,916	5	,502
	Exthet X	,219	6	,200*	,971	6	,899
MELAST	Supreme	,185	5	,200*	,902	5	,422
	Grandio	,224	5	,200*	,875	5	,286
	Exthet X	,142	6	,200*	,993	6	,994

*. This is a lower bound of the true significance.

a. Lilliefors Significance Correction

Test of Homogeneity of Variances

	Levene Statistic	df1	df2	Sig.
NANOD	,097	2	13	,908
MELAST	2,467	2	13	,124

ANOVA

		Sum of Squares	df	Mean Square	F	Sig.
NANOD	Between Groups	321246,5	2	160623,272	294,208	,000
	Within Groups	7097,359	13	545,951		
	Total	328343,9	15			
MELAST	Between Groups	195,765	2	97,883	97,384	,000
	Within Groups	13,067	13	1,005		
	Total	208,832	15			

NANOD

Tukey HSD[a,b]

		Subset for alpha = .05		
RESINA	N	1	2	3
Exthet X	6	392,9467		
Supreme	5		474,7960	
Grandio	5			727,0320
Sig.		1,000	1,000	1,000

Means for groups in homogeneous subsets are displayed.

a. Uses Harmonic Mean Sample Size = 5,294.

b. The group sizes are unequal. The harmonic mean of the group sizes is used. Type I error levels are not guaranteed.

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Comissão Científica e de Ética
Faculdade da Odontologia da PUCRS

Porto Alegre 28 de maio de 2008

O Projeto de: Pesquisa

Protocolado sob nº:	0031/08
Intitulado:	Comparação de propriedades mecânicas entre resinas compostas nanohíbridas e nanoparticulada
Pesquisador Responsável:	Prof. Dr. Eduardo Gonçalves Mota
Pesquisadores Associados	Rogério Simões Rosa
Nível:	Doutorado

Foi ***aprovado*** pela Comissão Científica e de Ética da Faculdade de Odontologia da PUCRS em *28 de maio de 2008.*

Prof. Dr. Eraldo Luiz Batista Júnior
Presidente da Comissão Científica e de Ética da
Faculdade de Odontologia da PUCRS

Av. Ipiranga, 6681, Prédio 06 sala 209
Porto Alegre /RS – Brasil – Cx. Postal:1429
90619-900

Fone/Fax: (51) 3320-3538
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